

Comparison of X-ray -  $e^-$  Coincidence and Noncoincidence  
Conversion Electron Mössbauer Spectra

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The performance of the X- $e^-$  coincidence technique for recording depth sensitive  $^{57}\text{Fe}$  CEMS spectra was investigated.

### 1. Introduction

Conversion Electron Mössbauer Spectroscopy /CEMS/ is increasingly being used, as a nondestructive depth integrated or depth sensitive tool, for the analysis of the surface layers. The depth integrated technique is suitable for the identification of the chemical compounds comprising surface layers. In this technique almost all back scattered electrons are detected by an  $\text{Ne}/\text{CH}_4$  flow proportional counter. For the  $^{57}\text{Fe}$  isotope, these are the L-shell /13.6 keV/ and K-shell /7.3 keV/ conversion electrons as well as the K-LL /5.5 keV/ and L-L $\bar{L}$  /0.53 keV/ Auger electrons. The intensity ratios and the energy distributions of these originally monoenergetic beams of electrons are changed during the electron interaction in the scatterer. The contribution of each surface layer to the depth integrated CEMS spectrum originates from a few electron beams of different energies and thus of different penetration depth. This complicates interpretation of the depth integrated CEMS spectrum.

In a more detailed surface analysis the depth sensitive technique is used. It is based on knowledge of an electron energy distribution as a function of depth in the scatterer. The K-shell conversion electron line is usually selected by application of an electron spectrometer and the correlation between electron energy loss and the electron depth of the origin is utilized in depth profiling Mössbauer studies. The depth

sensitive technique is time consuming due to very low transmission of an electron spectrometer. This limits wide applications of that technique.

The use of the He/CH<sub>4</sub> flow proportional counter and the X-e<sup>-</sup> coincidence technique in CEMS studies offers some sort of compromise between the depth resolution and the spectra storage time. The purpose of this paper is to investigate the performance of the He/CH<sub>4</sub> flow proportional counter and the X-e<sup>-</sup> coincidence technique for recording depth sensitive <sup>57</sup>Fe CEMS spectra.

## 2. Experimental technique

The He/CH<sub>4</sub> proportional counter has widely been used in depth integrated CEMS investigations. This is mainly due to its high counting efficiency and 2π geometry for scattered electrons. Up to now the energy resolution of the He/CH<sub>4</sub> proportional counter was thought to be very poor and therefore it was not taken into account in depth integrated CEMS investigations. However, the energy resolution of the He/CH<sub>4</sub> flow proportional counter is about 17.7% for the 6.36 keV electrons /1/. This allows selecting at least four nonoverlapping energy intervals within the 0 to 7.3 keV energy range and studying depth sensitivity of the CEMS spectra /2/. Even if depth sensitivity of the counter is not sufficiently good for depth profiling CEMS investigations one should know how the experimental conditions, such as setting of discrimination levels or instability of gas stream, effect the results.

In order to improve depth sensitivity of the He/CH<sub>4</sub> flow proportional counter one should eliminate all contributions but the K-shell conversion-electron one to the CEMS spectrum. This may be done, without a large decrease in the counting rate, by application of the X-e<sup>-</sup> coincidence technique. Our experiments were performed at room temperature using a <sup>57</sup>Co/Gr/ source and a constant acceleration Westbauer spectrometer. Two Westbauer scatterers were used in the experiments: a 10.7% <sup>57</sup>Fe enriched stainless steel foil and 90.6% <sup>57</sup>Fe enriched hematite and

magnetite layers on an iron substrate. A duplex layer of hematite and magnetite was produced by oxidizing an iron foil at 623 K in 1013 hPa of air for 3.5 hours. Hematite was the top layer, magnetite was the second layer and the iron substrate was the third layer of the investigated scatterer. The iron foil was neither polished nor annealed prior to oxidizing. An one-wire He/CH<sub>4</sub> flow proportional counter was used for detection of electrons emerging from a scatterer surface. Conversion X-rays (6.47 keV), produced inside the scatterer simultaneously with the K-shell conversion electrons (7.3 keV), were registered by an Ar/CH<sub>4</sub> proportional counter which was placed just behind the resonant scatterer. The X-ray pulses were used for coincidence purposes (1, 2).

### 3. Results and discussion

In order to show the performance of the X-e<sup>-</sup> coincidence technique for GEMS investigations the nuclear resonance contribution to the electron pulse-height spectrum (of the stainless steel scatterer) was determined (Fig. 1) with application (A) and without application (B) of the coincidence technique. The coincidence spectrum (A) originates only from the K-shell conversion electrons. The intensity of the K-shell electron beam increases with the increase in electron energy (decrease in depth) what should be expected. Apart from the K-shell conversion electrons, the L-shell conversion electrons and the K-L<sub>23</sub> Auger electrons are seen in the noncoincidence spectrum B.

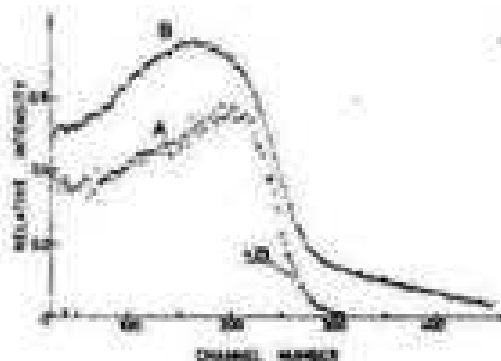


Fig. 1. The nuclear resonance contribution to the beam of back scattered electrons determined for the <sup>57</sup>Fe enriched stainless steel scatterer with (A) and without (B) application of the X-e<sup>-</sup> coincidence technique.

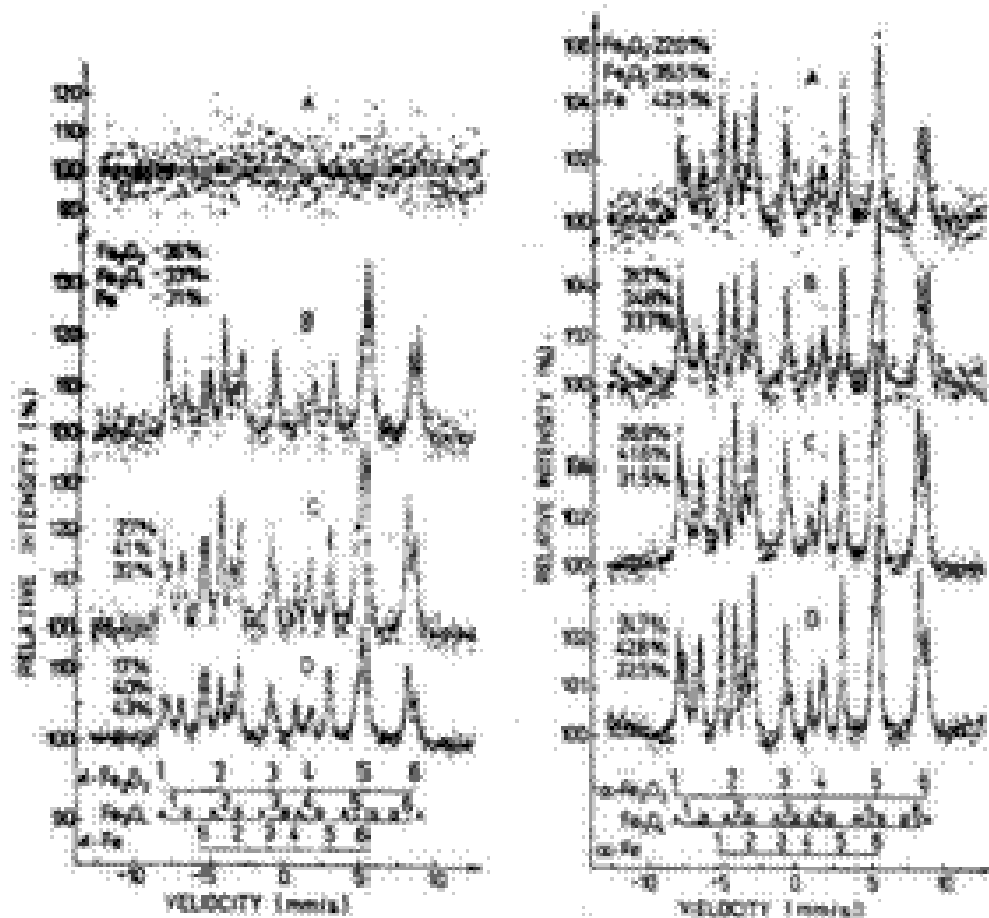


Fig.2. The coincidence /left/ and non-coincidence /right/ GEM spectra simultaneously recorded for the upper /A/, two intermediate /B and C/ and lower /D/ energy intervals of electrons emerging from the duplex layer of hematite and magnetite on the iron substrate.

In study of the depth sensitivity of the coincidence and non-coincidence GEM spectra we used the resonant scatterer composed of the duplex layer of hematite and magnetite on an iron substrate. Eight /four coincidence and the corresponding four non-coincidence/ GEM spectra were simultaneously recorded /Fig. 2/ for four energy intervals of the electron pulse-height spectrum. The A spectra were recorded for electrons of highest

outer sextet corresponds to the hematite layer /top layer/, the inner one to the iron substrate /third layer/ and the remaining two sextets are due to the A and B sublattices of the magnetite layer /second layer/. The positions of the peaks arising from various phases present and percentages of the total spectrum area contributed by each phase are indicated in Fig. 2. From their comparison the depth sensitivity of both coincidence and non-coincidence CEMS spectra is evident. The depth sensitivity is also visualized by changes of line amplitude ratios of various phases. The top hematite phase is most pronounced in the B coincidence spectrum while the deepest Fe phase is the D one what should be expected. The L-shell conversion electrons and the K-L Auger electrons noticeable influence the non-coincidence CEMS spectra. The iron contribution to the non-coincidence spectrum A is the largest one while the hematite contribution is the smallest one. This CEMS spectrum originates mainly from the L-shell conversion electrons. There is no corresponding one recorded by the coincidence technique.

In conclusion, both the  $X\text{-}\alpha^+$  coincidence and non-coincidence CEMS spectra recorded with the  $\text{He/CH}_4$  flow proportional counter are depth sensitive. This property should be taken into account in the depth integrated investigations or may be utilized in the preliminary depth sensitive surface studies. The  $X\text{-}\alpha^+$  coincidence technique improves depth sensitivity of CEMS spectra recorded with the  $\text{He/CH}_4$  flow proportional counter.

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#### References

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