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ANGLE SENSITIVE AND DEPTH SELECTIVE CEMS SPECTRA

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It is shown that a pronounced depth selectivity of CEMS spectra, recorded by the use of a helium-flow proportional counter, can be achieved both by selecting the angle of the incident γ-ray beam and by selecting the energy of the detected electrons.

1. Introduction

Iron 57 conversion electron Mössbauer spectroscopy (CEMS) is increasingly being used, as a nondestructive depth integrated or depth sensitive (DCEMS) tool, for the analysis of surface layers. These properties of the method are determined by a very short penetration range in solids (~ 100 nm) of low energy electrons (~ 7 keV) and especially by a successive reduction in their kinetic energy along penetration paths from the origin to a scatterer surface. Detection of all electrons emerging from the scatterer surface, regardless of their kinetic energy, leads to the depth integrated CEMS spectra. For storing the depth selective CEMS spectra one has to detect electrons of selected energies. The limited use of a helium-flow proportional counter for this purpose has been demonstrated [1–4] but the energy selection is usually carried out by an electron spectrometer. In the depth selective technique the correlation between the electron energy loss and the depth of the electron origin is utilized. The percentage contribution of a top layer of a scatterer to a total spectrum area can be enhanced by recording a CEMS spectrum at glancing angle of an incident gamma ray beam.

The purpose of this work was to study the performance of a helium-methane flow proportional counter in preliminary DCEMS investigations. Although the counter has not good enough energy resolution for sophisticated DCEMS studies, its luminocity is very large; it can accept electrons scattered into 2π solid angle.

2. Experimental

^{57}Fe conversion electron Mössbauer spectra were recorded at room temperature for a duplex layer of haematite and magnetite on a metallic iron substrate. All three layers were 90.6% enriched in ^{57}Fe. Haematite was the top layer,

magnetite was the second layer and metallic iron was the third layer. A constant acceleration Mössbauer spectrometer of the Polon type was equipped with a 50 mCi ^{57}Co (Cr) source and was used in the experiments. The gamma ray beam was always very well collimated by Soller's slits. Electrons emerging from the scatterer surface were detected with a thin He/CH_4-flow proportional counter. Its energy resolution for 6.3 keV electrons was 17.7%. In some experiments the K-shell conversion electrons (7.3 keV) were selected from an electron beam by the X-ray-e^- coincidence technique [5]. Electrons, with proper energies for DCEMS investigations, were selected by discrimination of electric pulse amplitudes with the single channel analyzers. Up to eight DCEMS spectra were simultaneously stored in some experiments; four coincidence and four non-coincidence ones. Some angle dependent CEMS spectra were numerically simulated and experimentally verified. The Mössbauer scattering spectra were decomposed into Lorentzian lines by the least squares computer procedure.

3. Results and discussion

The depth sensitivity of the method can be seen even in the shape of the on-resonance electron pulse-hight spectrum recorded with the He/CH_4 counter for a 97.9% ^{57}Fe enriched stainless steel scatterer. Two such spectra are shown in fig. 1. Spectrum B reflects all electrons emerging from the scatterer surface: the K-shell (7.3 keV) and L-shell (13.6 keV) conversion electrons as well as the K-LL (5.5 keV) and L-MM (0.53 keV) Auger electrons. Spectrum A represents only the K-shell conversion electrons which were separated from an electron beam by

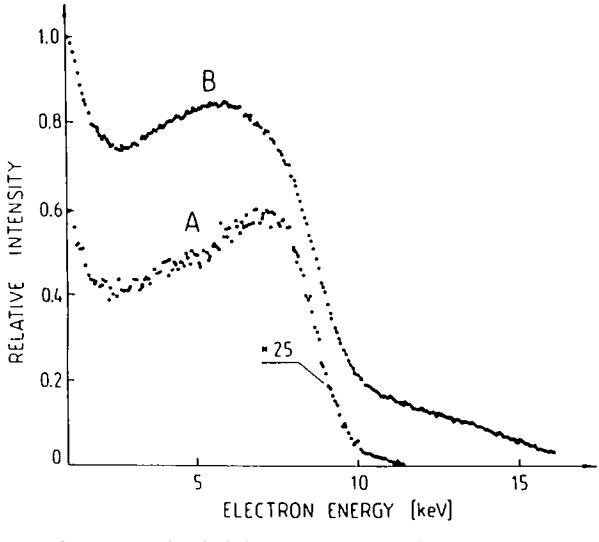

Fig. 1. The on-resonance electron pulse-height spectra recorded for a stainless steel scatterer with (A) and without (B) application of the X-ray–e^- coincidence technique.

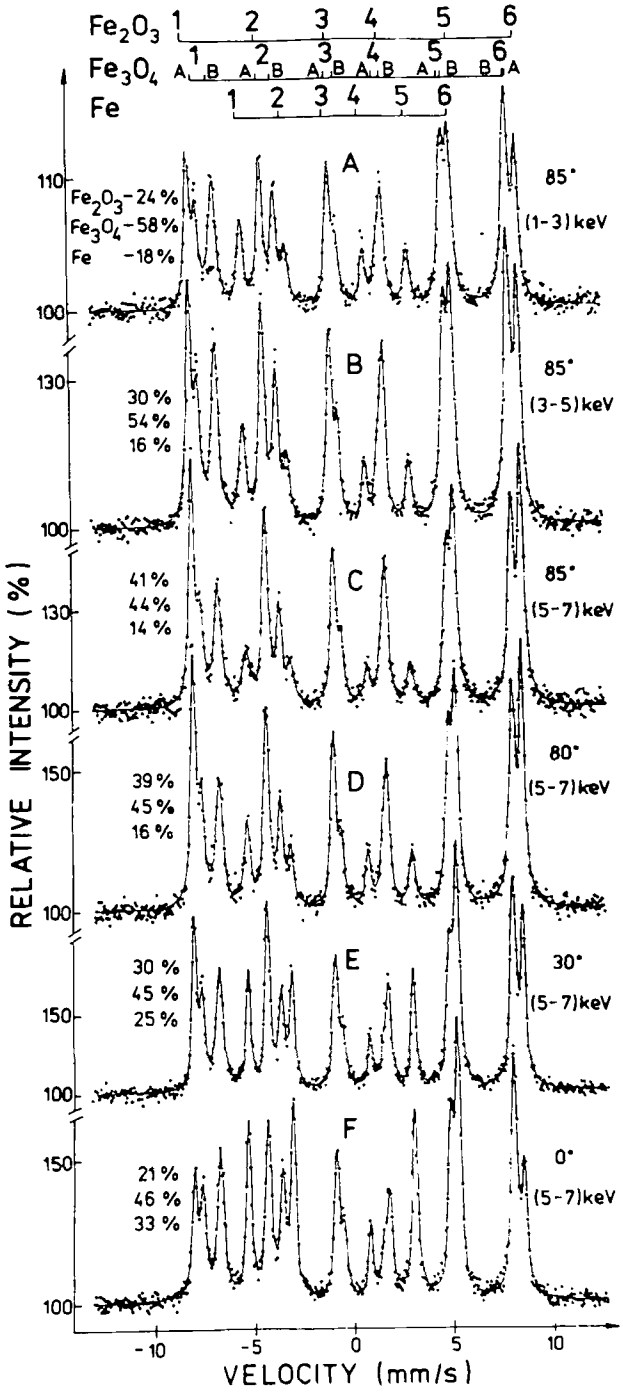

Fig. 2. The depth selective and angle sensitive CEMS spectra: A to C–recorded by registering electrons of selected energies, C to F–recorded at selected angles of incidence.

application of the X-ray–e^- coincidence technique. It is worth noticing that the intensity of the K-shell electron beam (A) monotonously increases with increase in electron energy (decrease in depth), as would be.

The depth selective and angle sensitive CEMS spectra, recorded for the duplex layer of haematite and magnetite on the metallic iron substrate using the He/CH_4 counter, are shown in fig. 2. The selected electron energy intervals, the angles of incident gamma rays relative to the normal to the scatterer surface and

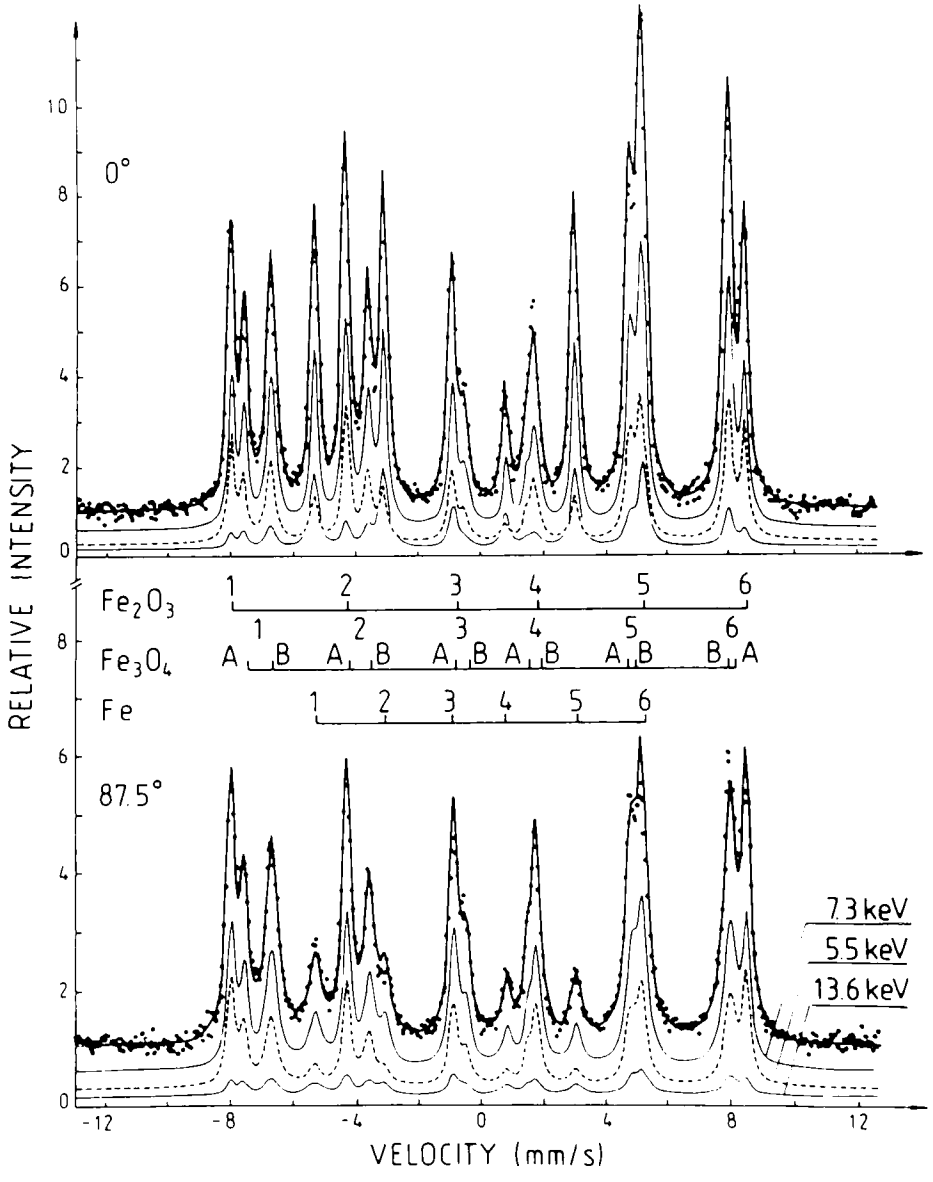

Fig. 3. Some of the depth integrated CEMS spectra with their conversion and Auger electron contributions.

the percentages of the total spectrum area contributed by each phase are indicated in fig. 2. The energy and angle dependences of the spectra are clearly vizualized. Contrary to the deepest iron phase the top haematite phase is most pronounced in spectrum C, which was recorded at largest incident angle for electrons of highest energy. Application of the X-ray–e^- coincidence technique [4,5] for selection of the K-shell conversion electrons improves the depth sensitivity of CEMS spectra. The depth integrated CEMS spectra recorded for our sandwich type scatterer are also angle dependent. The serie, recorded at selected angles of incidence (0°, 30°, 60°, 80°, 85° and 87.5°), is consistently described (fig. 3) by the theoretical lines which were calculated under the assumption of exponential attenuation of re-emited conversion electrons (7.3 keV and 13.6 keV) and Auger electrons (5.5 keV). The best agreement between the simulated spectra and the experimental ones was obtained for the layer thicknesses: 48 nm (haematite) and 192 nm (magnetite). The numerical values of the electron beam attenuation coefficients were calculated from an empirical formula [6].

In conclusion, the ^{57}Fe CEMS spectra recorded with the He/CH_4 flow proportional counter are sensitive both to the angle of the incident ray beam and to the energy of the electrons selected. These properties should be taken into account in depth integrated investigations or may be utilized in preliminary depth selective surface studies. The use of the He/CH_4 flow proportional counter and the X-ray–e^- coincidence technique in the depth selective CEMS studies offers some sort of compromise between depth resolution and spectra storage time.

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