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Depth Selective Mossbauer Analysis on a Barium Hexaferrite Single Crystal with a New UHV Orange Spectrometer

B. Stahl, R. Gellert, A. Kamzin*, G. Klingelhöfer and E. Kankeleit

Institut für Kernphysik, Schlossgartenstr. 9, TH Darmstadt, 64289 Darmstadt, Germany

* Laboratory of Magnetism, A.F. Ioffe Physico-Technical Institute RAS, Politekhnikeskaya 26, St. Petersburg 194021, Russia

Abstract. To study the hyperfine interaction as function of depth in a solid sample a new ultrahigh vacuum magnetic spectrometer has been designed and built for depth selective Mossbauer spectroscopy. To cope with the low intensity of emitted electrons in such an experiment the magnetic spectrometer of the orange type combines a high transmission of $\approx 20\%$ of 4π with an adjustable energy resolution in the order of 0.2 to 2 %. Furthermore, the focusing has been optimised to allow the simultaneous registration of events in an energy band of $\pm 4\%$ of the selected energy. The multi channel detector will provide 8 azimuthal and 5 energy channels.

First ^{57}Fe -Mossbauer experiments on magnetic single crystals have been performed to look for a variation of the magnetic hyperfine field with depth in the sample. The feasibility of this non-destructive depth selective analysis in studying surface magnetism of bulk samples has been demonstrated on a non-enriched $\text{BaFe}_{12}\text{O}_{19}$ single crystal.

I. Introduction

Depth selective Conversion Electron Mossbauer Spectroscopy (DCEMS) is a non-destructive method that provides information about hyperfine fields as function of depth in the sample in a sensitive volume of 0 - 300 nm in the case of ^{57}Fe . The depth resolution at the very surface is in the range of ≤ 5 nm depending on the statistical quality of the Mossbauer spectra and the energy resolution of the electron spectrometer. The energy loss of conversion and Auger electrons is related to a depth information by a deconvolution procedure that is based on data from a Monte Carlo simulation of the electron transport in the sample [1,2].

II. The Orange Type Magnetic Spectrometer

As count rates are very much limited by Mossbauer source strength and enrichment in the Mossbauer isotope in the absorber an electron spectrometer of high luminosity is required. For DCEMS the focusing of a high transmission orange type (21 % of 4π) magnetic spectrometer [2-4] was optimised to allow the simultaneous measurement of Mossbauer spectra in an electron energy interval of 8% of the energy setting in 5 non-equidistant channels by the use of a multi channel detector. As the highest accepted energy by the spectrometer is at least 45 keV experiments with several Mossbauer isotopes are possible (for example ^{57}Fe 14 keV, ^{119}Sn 20 keV, ^{151}Eu 22 keV, ^{180}W 34 keV/ 43 keV, ^{182}W 31 keV, ^{180}Hf 28 keV/ 43 keV). The energy resolution of the UHV orange spectrometer is in the range of 0.2- 2 % depending on the size of the sample. Experiments at low temperature are possible as the ultrahigh vacuum of less than 10^{-8} mbar reduces surface adsorbates on the sample.

III. Measurements on Barium Hexaferrite

The idea is to study magnetic properties of bulk samples as a function of depth and temperature. Due to the symmetry break at the surface different magnetic properties may occur in comparison to the bulk.

A first ^{57}Fe depth selective Mossbauer experiment on a $\text{BaFe}_{12}\text{O}_{19}$ single crystal has been performed.

Fig. 1 shows the measurements on a $\text{BaFe}_{12}\text{O}_{19}$ single crystal with natural abundance of 2 % in the isotope ^{57}Fe . Mossbauer spectra have been recorded at electron energies between 6.9 - 7.4 keV with a resolution of 2 %, covering a depth of approximately 0 - 50 nm in the sample. The spectrum at 7.39 keV is most sensitive to a surface layer of 0 - 10 nm, that at 6.9 keV to a layer of 0 - 40 nm. The precise weight function is plotted to the right of each Mossbauer spectrum, respectively.

The γ -ray direction is given by the measuring geometry and is nearly perpendicular to the surface of the sample: the diameter of the sample is 8 mm, that of the $^{57}\text{Co}(\text{Rh})$ source 4 mm and their distance 10.5 mm. The perfect orientation of the magnetic hyperfine field perpendicular to the surface is clearly indicated by the absence of those lines at $\approx \pm 4$ mm/s ($\Delta m = 0$) that are normally present in random orientation. The small contribution that is revealed by a least square fit is consistent with the geometry of sample and γ -source.

The analysis of the five Mossbauer spectra shows no variation in relative line intensities with different electron energies. Due to the good quality of the measured spectra this reveals that a possible reorientation of the magnetic field direction is limited to a surface layer of less than 10 nm. For previous results see ref. [5,6].

It should be mentioned that in this kind of experiment the transmission and not the luminosity of the spectrometer is crucial to the count rate as the solid angle for the γ -rays is limited by the maximum angle of incidence which one would allow to occur. In this respect the orange spectrometer clearly shows its superiority to electrostatic analyzers.

IV. Outlook

This first depth selective Mossbauer experiment on a non-enriched sample in combination with a new ultrahigh vacuum electron spectrometer with high transmission shows the feasibility of the non-destructive method in studying surface magnetism of bulk samples.

The above measurements have been performed in sequence with a single channeltron detector. The quality of the spectra is already sufficient to reveal the depth dependence of hyperfine parameters over an interval of 0 - 50 nm in the sample which were shown to be constant in the example of a barium hexaferrite single crystal. The statistical quality will still be improved in future experiments when the multi channel detection system will have been installed in the orange spectrometer using the dispersion for a simultaneous registration of events in an energy band of ± 4 %. Furthermore, temperature dependent measurements are in preparation.

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Figure 1: ^{57}Fe -Mossbauer spectra recorded at electron energies between 6.9 - 7.4 keV at the K conversion edge of 7.3 keV. The $\text{BaFe}_{12}\text{O}_{19}$ single crystal was 8 mm in diameter and has the natural abundance of ≈ 2 % in the isotope ^{57}Fe . To the right of each Mossbauer spectrum the probability density with respect to depth is plotted for the chosen electron energy considering a spectrometer resolution of 2 %.