

# IRON HYPERFINE FIELDS AT THE FE-AU INTERFACE

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## 1. Abstract

We studied the effect of interfaces structure in <sup>57</sup>Fe/Au thin layers on iron hyperfine parameters using Mössbauer spectroscopy. Four samples of bilayers (<sup>57</sup>Fe covered by Au) were prepared on MgO substrate by pulsed laser deposition (PLD) with varying <sup>57</sup>Fe layer thickness (~10 – 95 nm), covered by Au layer with constant thickness of ~8.7 nm. Mössbauer spectra were acquired in transmission and conversion electron (CEMS) modes. The hyperfine parameters of  $\alpha$ -Fe layer, Fe/Au and MgO/Fe interfaces were obtained.

## 2. Introduction

The primary aim was the preparation of stable thin <sup>57</sup>Fe layers for fast calibration of Conversion Electron Mössbauer spectroscopy. The iron layer was protected by gold overlayer to prevent its surface oxidation or degradation. It is, however, necessary to study the possible influence of the covering gold layer on the hyperfine parameters (especially hyperfine field  $B_{\text{HF}}$ ) of iron. At room temperature iron possesses  $\alpha$ -phase (BCC) and solubility of gold in this iron phase is very low, up to ~3 wt.% [1]. But at the interface between Fe and Au a layer of different phase(s) can be formed, e.g., FCC alloy Au/Fe. In the FCC structure of Au/Fe the solubility of Fe in is Au much higher [2]. The presence of an Au atom in the surrounding of an iron one can radically change the value of  $B_{\text{HF}}$  and these samples could not thus be used for the calibration of Mössbauer spectrometers.

## 3. Experimental

### 3.1. Sample preparation

Iron/gold bilayers were deposited by PLD (Fig.1) on MgO single-crystalline(100) substrate using Nd:YAG laser operating at 266 nm wavelength with the repetition rate of 10 Hz and providing energy of 130 mJ in 5 ns light pulses. The laser beam was focused on the target by means of quartz lens to a spot with 3 mm diameter. The laser energy density on targets was ~2.6 J/cm<sup>2</sup>.

Ultra-high purity <sup>57</sup>Fe and Au targets were ablated by laser pulses. The distance between the substrate and the target was ~55 mm. The substrate temperature was 100 °C and the substrate was outgassed before deposition for 1 hour. The base pressure in the deposition chamber was better than 10<sup>-5</sup> Pa. The time for growing of 1 monolayer of residual gasses at these conditions is approx. 36 sec. Final thickness of iron layer was monitored by the number of laser pulses and measured using spectral ellipsometry on one sample. Parameters of the prepared samples are summarized in the Tab. 1.

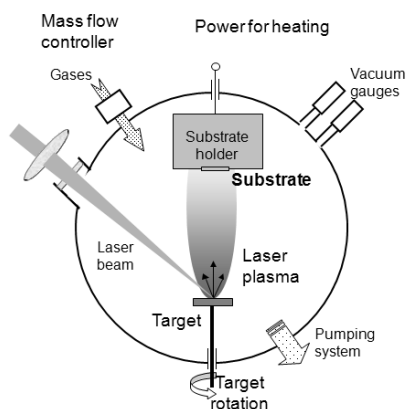


Fig. 1 The schematic experimental setup of PLD

Tab. 1 Parameters of the prepared samples

Sample	Thickness of Fe, calc.[nm]	Thickness of Fe (ellipsometry) [nm]	Number of Laser Pulses	Substrate (thickness 0,5 mm)
Au:57Fe_1	10.4	13.1	460	MgO
Au:57Fe_2	10.4	---	460	MgO
Au:57Fe_3	20.8	---	920	MgO
Au:57Fe_4	95	---	4200	MgO

To prevent contamination and oxidation the iron surface was covered by gold layer also by PLD by means of 200 laser pulses with thickness 8.7 nm (measured by spectral ellipsometry) with shorter delay after iron deposition.

### 3.2. Sample characterization

The samples were characterized by the scanning electron microscope Tescan Vega 5136XM in back-scattered electrons and analyzed using energy dispersion roentgen fluorescence analyzer Oxford/Inca. In the samples the substrate (MgO), iron and gold were only detected. On the surface of sample Au:57Fe\_1 a lot of spherical gold particles with the diameter up to 1 – 2 micrometers were present. This ball shaping of particles is typical for the thermally generated particles. It is possible that most of the gold on the surface of the sample Au:57Fe\_1 was in these round particles and did not create continuous surface layer. (Fig. 2)

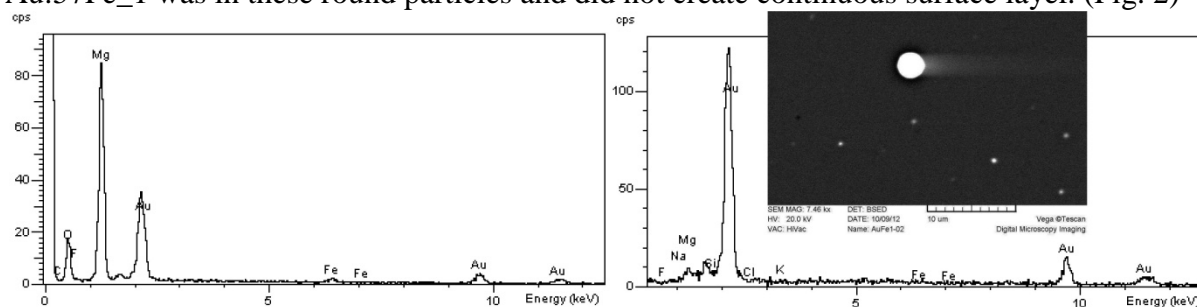


Fig.2 The EDX spectrum characterizing the composition of the sample in a smooth part of the surface (left), the EDX spectrum and the BSE picture of the spherical gold particles stuck on the surface (right)

### 3.3. Mössbauer measurements

Mössbauer spectra were acquired at room temperature (RT) in the transmission and conversion electron (CEMS) modes with constant acceleration of the  $^{57}\text{Co}(\text{Rh})$  source. The

calibration of velocity scale was performed with natural  $\alpha$ -Fe. The spectra were evaluated using the program CONFIT [4]. Mössbauer spectra are on the Fig. 3.

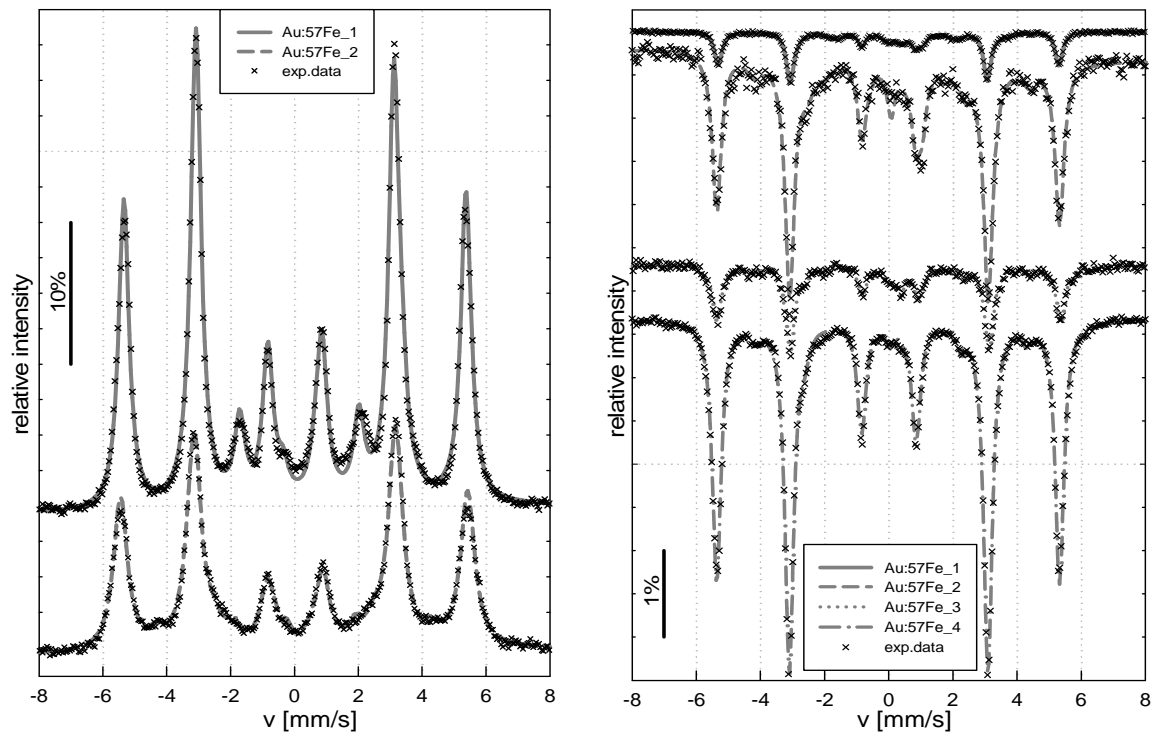


Fig. 3 Spectra of samples 1, 2 in CEMS (left) and samples 1-4 in transmission mode (right)

#### 4. Results and discussion

The parameters obtained from measured Mössbauer spectra are given in Tab. 2. The components with  $B_{HF} = 33.1 \pm 0.1$  T (and IS and QS  $\sim 0$  mm/s) correspond with  $\alpha$ -Fe are correctly and predominantly detected in all samples and in all arrangements of measurement.

In samples Au:57Fe\_1 and Au:57Fe\_2 components with  $B_{HF} = 20.0 \pm 0.4$  T (IS  $\sim 0,18 \pm 0.10$  mm/s, QS  $\sim 0,09 \pm 0.09$  mm/s) were detected. Hyperfine parameters of these components correspond with parameters for cementite ( $Fe_3C$ ) [5]. Formation of cementite is possible from organic residues and carbon-containing gases, present on the surface of the  $^{57}Fe$  target. The highest amount of cementite component was detected in sample (Au:57Fe\_1). In sample Au:57Fe\_2 the component related to the  $Fe_3C$  phase is detected in lower proportion in comparison with sample Au:57Fe\_1. In samples Au:57Fe\_3, 4, analyzed using transmission mode MS, this component is not detected. It corresponds to the gradual “cleaning” of surface of the  $^{57}Fe$  target from the carbon impurities.

The component with hyperfine parameters  $B_{HF} = 26.6 \pm 0.4$  T (IS  $\sim 0,01 \pm 0.06$  mm/s, QS  $\sim 0,15 \pm 0.09$  mm/s) is ascribed to the presence Au atoms in the surroundings of Fe atoms [6] in the interface Fe/Au layer. In the first sample Au:57Fe\_1 this component was not detected, because the gold layer was not properly created due to carbon and cementite in the iron layer. In samples Au:57Fe\_2, Au:57Fe\_3 and Au:57Fe\_4 the relative amount of this component decreases in correspondence with increasing iron layer thickness.

Doublets, identified in all samples with hyperfine parameters IS =  $0,47 \pm 0.11$  mm/s, QS  $\sim 1,0 \pm 0.10$  mm/s supposedly belong to Fe atoms diffused into the MgO substrate [7].

Singlets with IS  $\sim 0.38 \pm 0.05$  mm/s found in samples Au:57Fe\_1 and Au:57Fe\_3 in transmission mode are an artifact of our measuring equipment for extremely long expositions due to the presence of iron traces in the Al foil covering the detector window.

Tab. 2 Mössbauer parameters of the samples of Au-covered  $^{57}\text{Fe}$  layers on MgO

Sample	Möss. mode		$B_{\text{HF}}$ [T]	err. $B_{\text{HF}}$ [T]	IS [mm/s]	err.IS [mm/s]	QS [mm/s]	err.QS [mm/s]	rel.int. [%]
Au: $^{57}\text{Fe}_1$	CEMS	S1	33.0	0.2	0.01	0.03	-0.01	0.00	75
		S2	19.8	0.3	0.20	0.03	0.03	0.04	23
		D1	--	--	0.26	0.13	0.82	0.17	2
Au: $^{57}\text{Fe}_1$	transmis.	S1	33.0	0.2	0.00	0.03	0.00	0.03	71
		S2	20.2	0.3	0.27	0.04	0.14	0.05	17
		D1	--	--	0.54	0.07	1.07	0.08	7
		L1	--	--	0.39	0.09	--	--	5
Au: $^{57}\text{Fe}_2$	CEMS	S1	33.2	0.2	0.01	0.03	-0.02	0.03	76
		S2	20.1	0.5	0.13	0.05	0.00	0.03	5
		S3	26.6	0.3	0.06	0.04	0.02	0.05	18
		D1	--	--	0.44	0.19	1.10	0.15	1
Au: $^{57}\text{Fe}_2$	transmis.	S1	33.2	0.2	0.00	0.03	-0.03	0.03	71
		S2	19.9	0.3	-0.02	0.09	0.18	0.12	7
		S3	26.6	0.2	0.05	0.03	0.15	0.03	16
		D1	--	--	0.56	0.13	0.98	0.10	7
Au: $^{57}\text{Fe}_3$	transmis.	S1	33.3	0.2	0.02	0.03	-0.03	0.03	82
		S2	26.8	0.4	-0.04	0.05	0.18	0.07	8
		D1	--	--	0.54	0.11	1.03	0.09	5
		L1	--	--	0.37	0.14	--	--	5
Au: $^{57}\text{Fe}_4$	transmis.	S1	33.2	0.2	-0.01	0.03	-0.02	0.03	93
		S2	26.2	0.3	-0.04	0.07	0.24	0.08	5
		D1	--	--	0.48	0.16	0.89	0.17	2

S – sextet, D – doublet, L – singlet, CEMS – Conversion Electron MS, transmis. – transmission MS,  $B_{\text{HF}}$  – hyperfine field, err. – error, rel. int. – relative intensity of the component in spectrum

## 5. Conclusions

Samples of thin layers of  $^{57}\text{Fe}$  covered by Au were prepared by PLD. In the samples the effect of MgO/Fe and Fe/Au interfaces was detected and  $\text{Fe}_3\text{C}$  phase was found, originating from the carbon contamination of the  $^{57}\text{Fe}$  target surface. For preparing good and stable calibration layers with only  $\alpha$ -iron component it is necessary to use a different way of preparation and to restrict the influence of interfaces.

## Acknowledgement

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