Chorzów, 08-01-2020

Final report for Palacky University Olomouc, Czech Republic

Main goal:

The room-temperature Mössbauer measurements of the original sample in order to test the presence of superparamagnetic iron nanoparticles entrapped in the graphene oxide matrix.

Measurement:

Transmission ^{57}Fe Mössbauer spectrum was recorded using an MS96 Mössbauer spectrometer operating in a constant acceleration mode and equipped with a ^{57}Co:Rh source with activity of ~50mCi. The spectrometer was calibrated at room temperature with 30 μm thick α-Fe foil. The time of the measurement was equal to about 1001 hours, which is more than 41 days. However, the effect was smaller than 0.1% and even after such a long time of the measurement the quality of the spectrum was not very good. The numerical analysis of the Mössbauer spectrum was performed with the use of the MossWinn program.

Results

The Mössbauer spectrum and the fitted components for the investigated sample are presented in Figure 1 and obtained hyperfine parameters for each component are listed in Table 1. The spectrum was fitted as a superposition of three components, which were singlet, doublet and sextet. These three components indicate presence of three iron-containing fractions. The relative area of the singlet is about 16%. The value of isomer shift obtained for this component is close to zero (Table 1) , which indicates presence of the ultrafine α-Fe nanoparticles in the investigated sample, which are in a superparamagnetic state at room temperature. The hyperfine parameters of the doublet are connected with presence of ferric ions in the sample with contribution of ~21%. The third component visible on the Mössbauer spectrum is a sextet with hyperfine magnetic field of 32.6T and contribution of about 63%. This sextet can also correspond to α-Fe but presence of this magnetically ordered phase will be a result of the magnetic blocking of some α-Fe nanoparticles.

To sum up, my measurement supports the original finding in Tucek et al, Nature Communication (2016) 7, 12879 (DOI: 10.1038/ncomms12879) and the related correction (DOI: 10.1038/s41467-019-10702-2). I take into account that the sample is about six years old and stored in a lab with air access, which can significantly influence contributions of iron-containing fractions.

Table 1. The Mössbauer hyperfine parameters of the investigated sample. Is – isomer shift, Qs – quadrupole splitting, H – hyperfine magnetic field, A – real area fraction of subspectra, FWHM – full line width at half maximum.

Component	Is (mm/s)	Qs (mm/s)	H (T)	A (%)	FWHM (mm/s)
Singlet	-0.018 ± 0.019	-	-	15.98	0.55
Doublet	0.276 ± 0.026	0.876 ± 0.039	-	21.18	
Sextet	0.113 ± 0.008	-	32.60 ± 0.09	62.84	

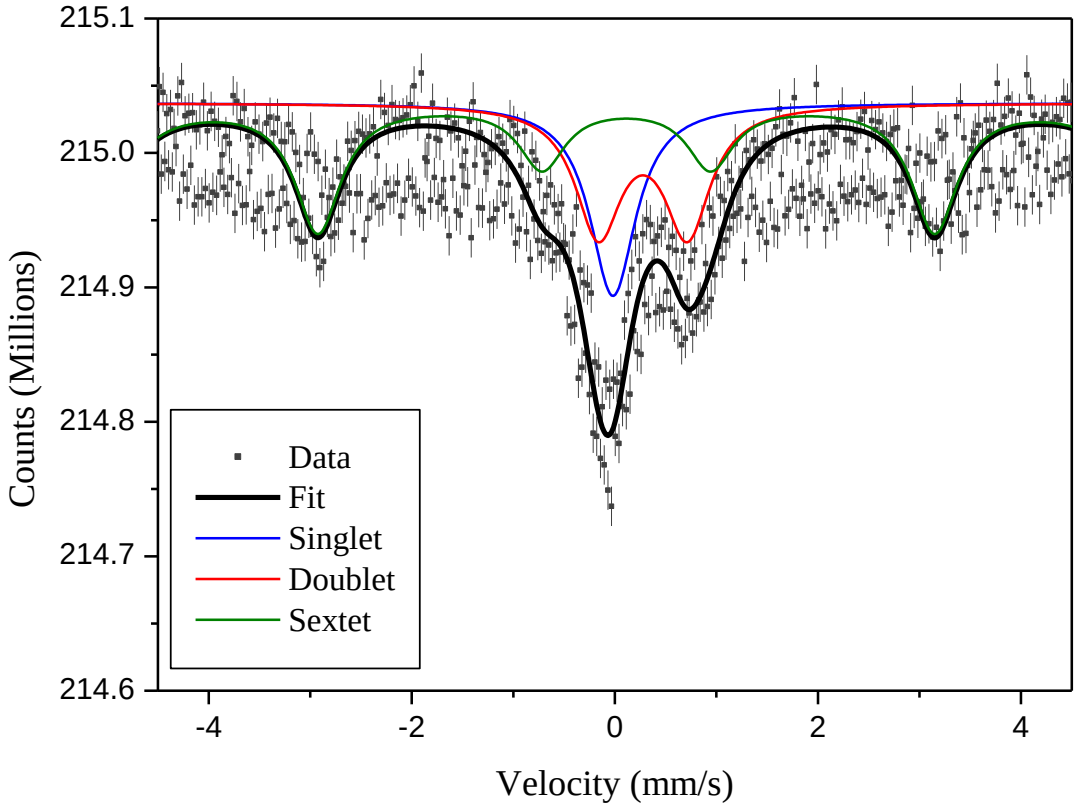

Figure 1. The room-temperature Mössbauer spectrum of the investigated sample. The fitted subspectra are presented on the spectrum.

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