Si-Shift

High-performant non-oriented electrical steels with a silicon content beyond today's limits:
new materials for an electrified future.

Deliverable D2.1 – Mössbauer Spectroscopy Protocol

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Abstract

Deliverable abstract
The Mössbauer spectroscopy protocol will deliver all the information necessary to perform an analyze Mössbauer experiments on Fe-Si samples, needed to study an order-disorder transition in Fe-Si system. The document describes the theoretical background of Mössbauer spectroscopy and details of the experimental setup. It defines the elements and materials necessary for the measurements. The main part of the protocol includes the description of preparing the spectrometer, calibration of the spectrometer transducer speed, software, sample measurement and analysis. As an illustration, the measurement and analysis of one sample is shown.

Contents

1 Introduction

As described in the Si-Shift proposal (1.2.2. Pillar 1: Material exploration), Mössbauer spectroscopy will be used as a tool to get information about local atomic arrangement in Fe-Si and Fe-Si-X samples. The method will complement *ab initio* calculations combined with machine learning and more conventional experimental characterization methods (e.g., X-ray diffraction, transmission electron microscopy TEM, etc.).

Mössbauer spectroscopy is a local technique (i.e., providing information at the atomic scale) using gamma radiation. It is especially suitable for iron-containing crystals because the naturally occurring ^{57}Fe isotope has ideal properties for this technique. Mössbauer spectroscopy provides a fingerprint spectrum with contributions from all types of surroundings of iron atoms in the alloy, at the atomic scale (sensitivity of a few atomic neighbour shells). It is possible to numerically deconvolute an experimental Mössbauer spectrum into single components, each reflecting a specific local configuration of atoms.

In the Si-Shift project, all the investigated samples are Fe-Si electrical steel in bulk form. At the stage of creating the Si-Shift proposal, it was established that it is impossible to produce thin sheets of these alloys with a thickness of 40-50 micrometers. Therefore, the Conversion Electron Mössbauer spectroscopy CEMS was established as a suitable technique for studies in the project.

2 Theoretical background of Conversion Electron Mössbauer spectroscopy CEMS

The effect of recoilless emission and absorption of γ radiation by the nuclei in solids discovered in 1957 by a German physicist, Rudolf L. Mössbauer became the basis for the development of a research technique called Mössbauer Spectroscopy (MS). The most popular Mössbauer nuclides are ^{57}Fe, ^{119}Sn, ^{151}Eu, ^{155}Gd, and ^{161}Dy because they exhibit the largest f factor, which means the probability of the Mössbauer effect.
It is the ratio of the recoilless photons that are emitted to the total number of photons, called also the Lamb-Mössbauer factor:

$$f = \exp\left(-k^2 < x^2 >\right), \tag{1.1}$$

where $k = 2\pi/\lambda$ is a wave vector and $<x^2>$ denotes the square average value of the amplitude of atom oscillations in the direction of the quantum momentum that is emitted. The value of the f factor strongly depends on the temperature and the energy of the transition of the nucleus from an excited to a ground state. Thus, the probability of the Mössbauer effect increases with a decrease in the measurement temperature and is larger for low energy transitions. In the case of isotope ^{57}Fe, the value of $f = 0.75$ at room-temperature for energy $E = 14.4$ keV. The iron is prevalent in nature and materials, and in all of the samples that contain iron, the relative concentration of ^{57}Fe isotope is equal to 2.17 %.

The main idea of the Mössbauer spectroscopy is the motion of the source of γ radiation (Figure 1).
The movement of the source relative to the absorber (the investigated sample) causes a change in the energy of γ quanta in accordance with the Doppler effect:

$$\Delta E = (V/c)\, E\gamma\,, \tag{1.2}$$

where $E\gamma$ is the energy of the γ quantum and V denotes the velocity of the source relative to the absorber. For γ radiation with an energy of 14.4 keV, the change of velocity by 1 mm/s causes a modulation of energy by 48 neV. This modulation allows the Mössbauer spectrum to be registered, i.e., the possible shifts and splitting of the absorption lines can be observed. The sensitivity of Mössbauer spectroscopy is one of the highest in experimental physics. The uncertainty of the determination of energy for ^{57}Fe is about 10^{-10} eV. The most important problem is to precisely determine the movement of the source with constant acceleration. The range of the source motion that is selected is dependent on the composition of the material.

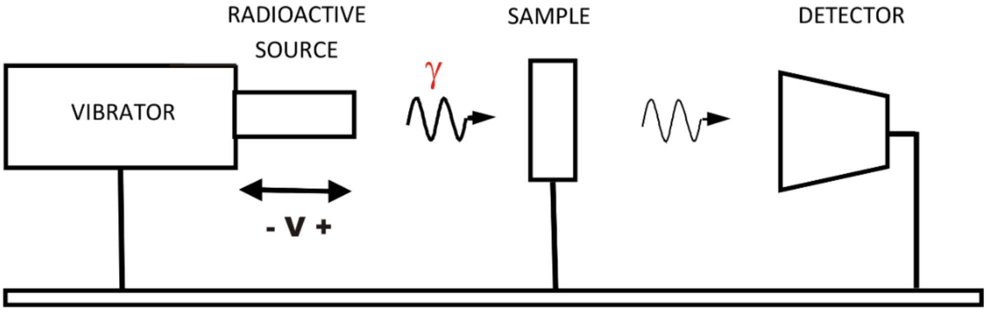


Figure 1. Schematic diagram of an experimental setup for Mössbauer spectroscopy in transmission geometry.

Two popular techniques are used to measure Mössbauer spectra. The first one is a transmission geometry in which the sample is placed between the mobile source and the detector (Figure 1). In this case, the absorber should be made of tens mg/cm^2 thick foil. The most effective technique for investigating thin films and surface layers of materials is Conversion Electron Mössbauer Spectroscopy – CEMS in which the sample is placed inside a conversion electrons counter that is filled with a mixture of gases.
In both techniques, transmission Mössbauer spectroscopy (TMS) and Conversion Electron Mössbauer Spectroscopy (CEMS), the same recoil-free nuclear resonance is the fundamental principle of the method; however, a different product of the resonance is utilized. Instead of 14.4 keV photons (in the case of TMS based on ^{57}Fe), 7.3 keV conversion electrons together with 5.4 and 6.2 keV Auger electrons are detected in CEMS technique (Figure 2).

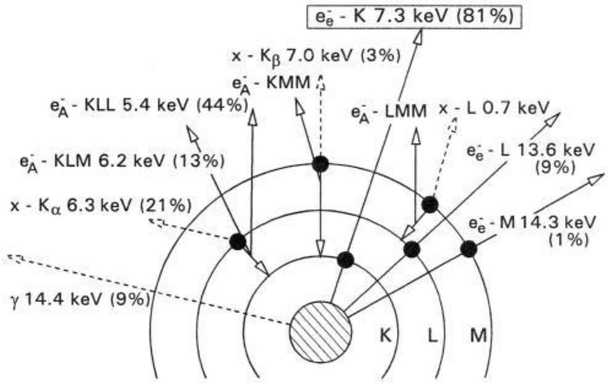


Figure 2. De-excitation of ^{57}Fe nucleus after resonance excitation [1].

In comparison with TMS, the CEMS technique does not require the minimum absorber thickness due to the much lower detection limit of CEMS. Moreover, the internal conversion process is much more probable than the emission of γ radiation. The fundamental principle of the CEMS measurement is presented in Figure 3. The penetration depth of 14.4 keV photons is about 40 μm, while that of 7.3 keV electrons reaches up to 350 nm. If the resonance occurs in bulk (Figure 3 a), all electrons are absorbed by a sample material, and they are no longer registered. However, when the resonance occurs in the upper surface layer, some electrons can escape and be detected (Figure 3 b) [2].

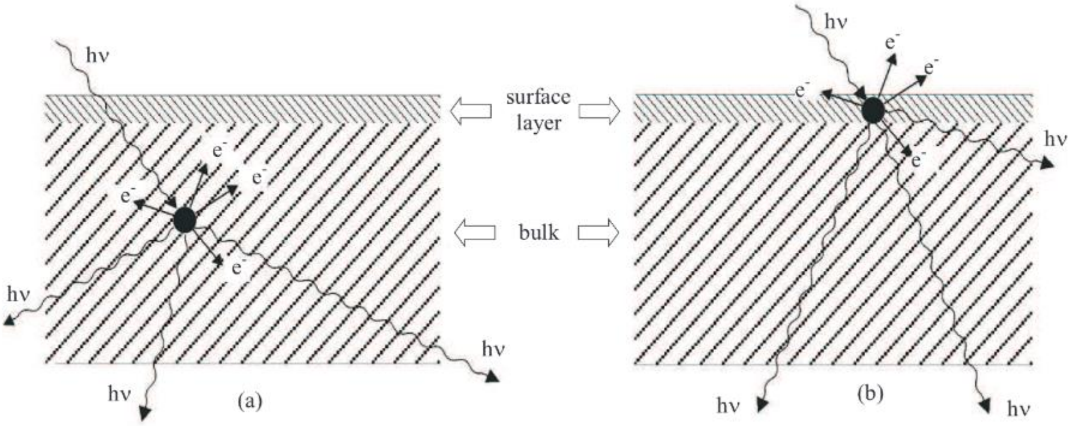


Figure 3. The principle of the CEMS measurement [2].

Mössbauer spectroscopy is a technique that can be used to investigate hyperfine interactions. These are interactions of the electromagnetic moments of the nuclear probe (i.e., ^{57}Fe isotope) with the electric and magnetic fields that originate from its electron shells as well as from the nearest neighborhood of the probe. A detailed description of hyperfine interactions can be found in many monographs and scientific articles (e.g., [3-5]). There are three the most important interactions: (i) the monopole electrostatic interaction between the positive charge of the atomic nucleus and the *s*-type electrons, (ii) the interaction between the quadrupole electric moment of the nucleus and the electric field gradient that is generated by the surrounding electrons and (iii) the interaction between the magnetic moment of the nucleus and the magnetic field at the nucleus site.

In general, all of the interactions mentioned above can occur simultaneously in the investigated material. The possible shifts and splitting of nuclear levels are reflected in the Mössbauer spectrum that is registered. The numerical fitting of the experimental spectrum together with the structural information, for example from the XRD patterns, allows the phase composition of the material to be recognized and its magnetic properties to be determined. The fitting procedure is performed until the best fitting parameter, Chisq (χ^2), is achieved. The hyperfine interaction parameters that are determined from the spectra are as follows: *IS* – the isomer shift relative to α-iron, *QS* – the quadrupole shift of the magnetically split spectra or the quadrupole splitting of the paramagnetic doublet, B_{hf} – hyperfine magnetic field, Γ – half width at half maximum of the spectral lines. All of the parameters are determined with an accuracy of about 0.01 mms^{-1}, which translates into an accuracy of the determination of the hyperfine interaction energy of the order of 10^{-10} eV.

3 Construction of a spectrometer

The block diagram of the CEMS spectrometer is presented in Figure 4.

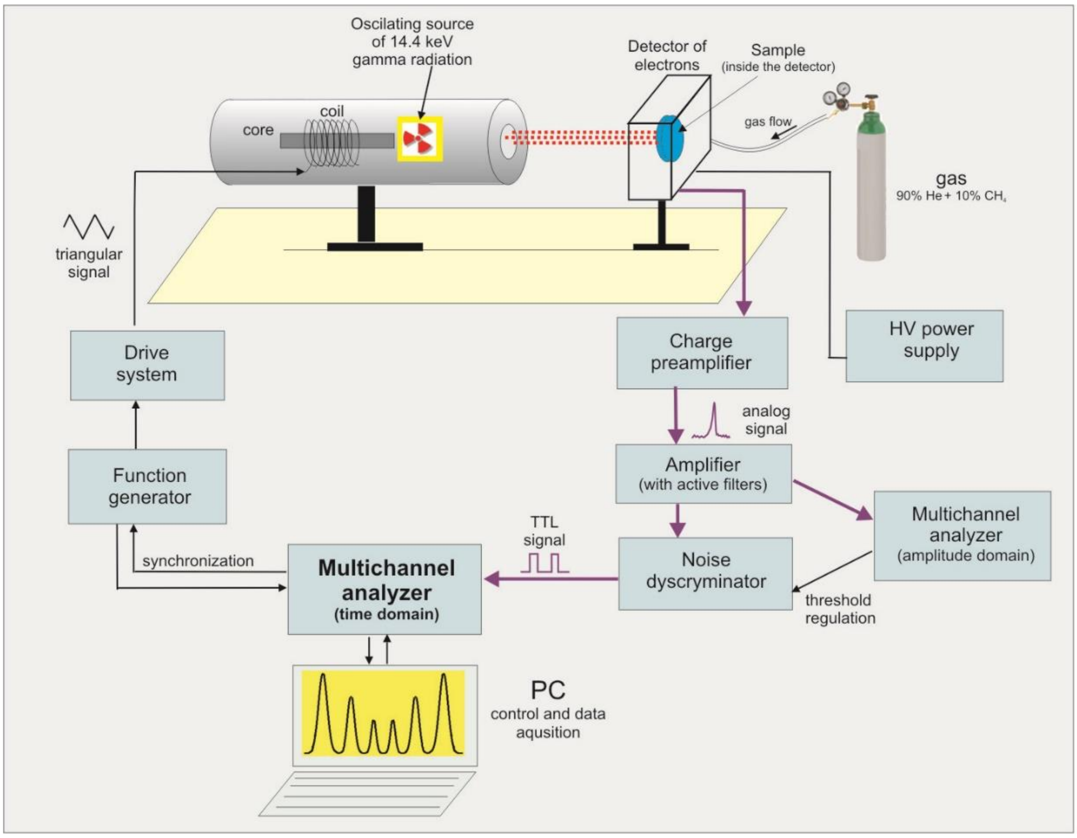


Figure 4. The CEMS measurement setup.

The heart of the system is a head with a moving radioactive source. The movement of the source causes a tiny modulation of energy of outgoing gamma quanta due to the Doppler effect. The source of 14.4 keV gamma radiation is ^{57}Co embedded in a cubic and paramagnetic matrix to avoid hyperfine structure of the source being overlaid on a hyperfine structure of the sample for investigation. The matrix usually is Rh or Cr. The source is attached to the end of the core of an electro-mechanic transducer. The coil of the transducer is supplied from a function generator. Various shapes of the signal can be used to drive the transducer, according the needs of a user; however, a triangular signal leading to the constant acceleration working mode of a spectrometer is used most frequently.

The sample for investigations is located inside the detector of electrons and grounded. High voltage from the power supply is applied between the sample and detection rods playing the role of a positive electrode. Thus, when the gamma quanta hitting the sample causes the emission of internal conversion electrons, they are accelerated towards positively charged detection rods. In this way electric pulses are generated as a consequence of conversion electron emission. Additionally, a very slow gas flow (~0.1 l/h) is necessary to ensure the stable work of the detector. The spike pulses coming from the detector are amplified and formed. To cut the low-amplitude electronic noise the discriminator is applied. The latter transforms spike pulses to TTL pulses that can be counted by a main multichannel analyzer working in a time domain (TMA). There is also another, auxiliary MA working in an amplitude domain (AMA). The amplitude spectrum of pulses obtained from the latter one can be used for discrimination threshold regulation by the user. The TMA is synchronized with the function generator driving the transducer thus the coming TTL pulses can be categorized into channels regarding the time of coming. After a calibration process, a specific velocity/energy can be ascribed to each time channel. In this way, a velocity/energy spectrum of gamma quanta causing the internal conversion phenomenon can be created on a PC.

4 Conversion electron counters

Because the probability of the emission of conversion electrons from the K shell is the largest (77 – 81 %), mainly electrons with low energy, i.e., 7.3 keV are detected. However, the detection of K-electrons is disturbed by an intensive gamma background. In order to suppress the gamma photons, a gas-flow proportional counter can be used in which helium is a counting gas with an admixture of methane as a quencher. The most important requirements for the counter are long-term stability and suppression of non-resonant background (with the minimum self-produced photoelectrons). Further parameters to consider are detector thickness, number of wires, wire diameter, gas mixture, gas-flow rate, and fixing the sample [6]. The scheme of a simple gas-flow proportional detector for CEMS measurements is presented in Figure 5.

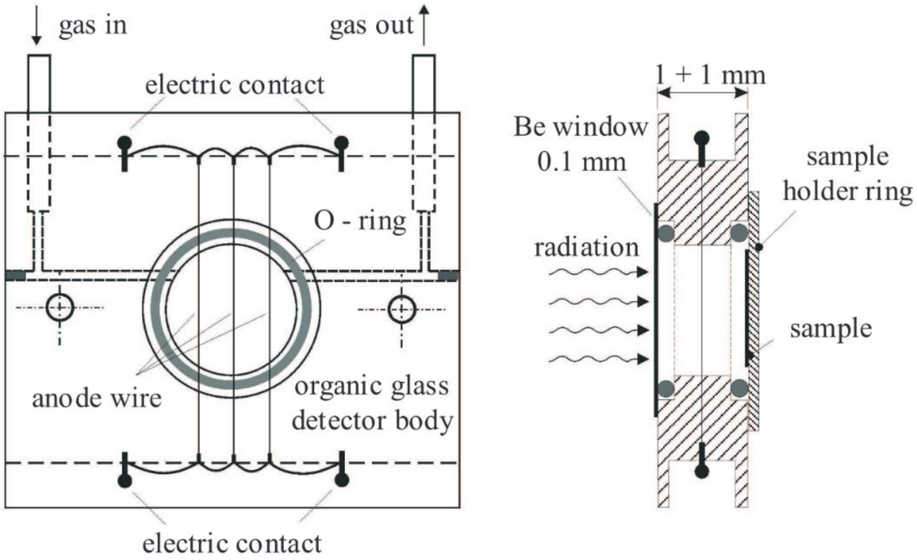


Figure 5. A simple gas-flow proportional detector for CEMS [2].

To suppress the gamma background, helium is used as a detection gas with an admixture of 4 – 12 % methane as a quenching gas. In the dependence of the methane concentration, there are various widths of the proportional band, i.e., between 1000 and 1360 V for 8 % of CH_4 and between 1145 – 1540 V for 15 % CH_4 [2]. The wider proportional band, the measurement is more stable, especially in the case of long-lasting measurements. The amount of gas flow between a few and a hundred cm^3 per hour is enough for good measurement.

As an example, a description for two conversion electron counters with different designs is presented as follows.

Counter LEK 2

The actual appearance of the LEK 2 detector is shown in Figure 6.

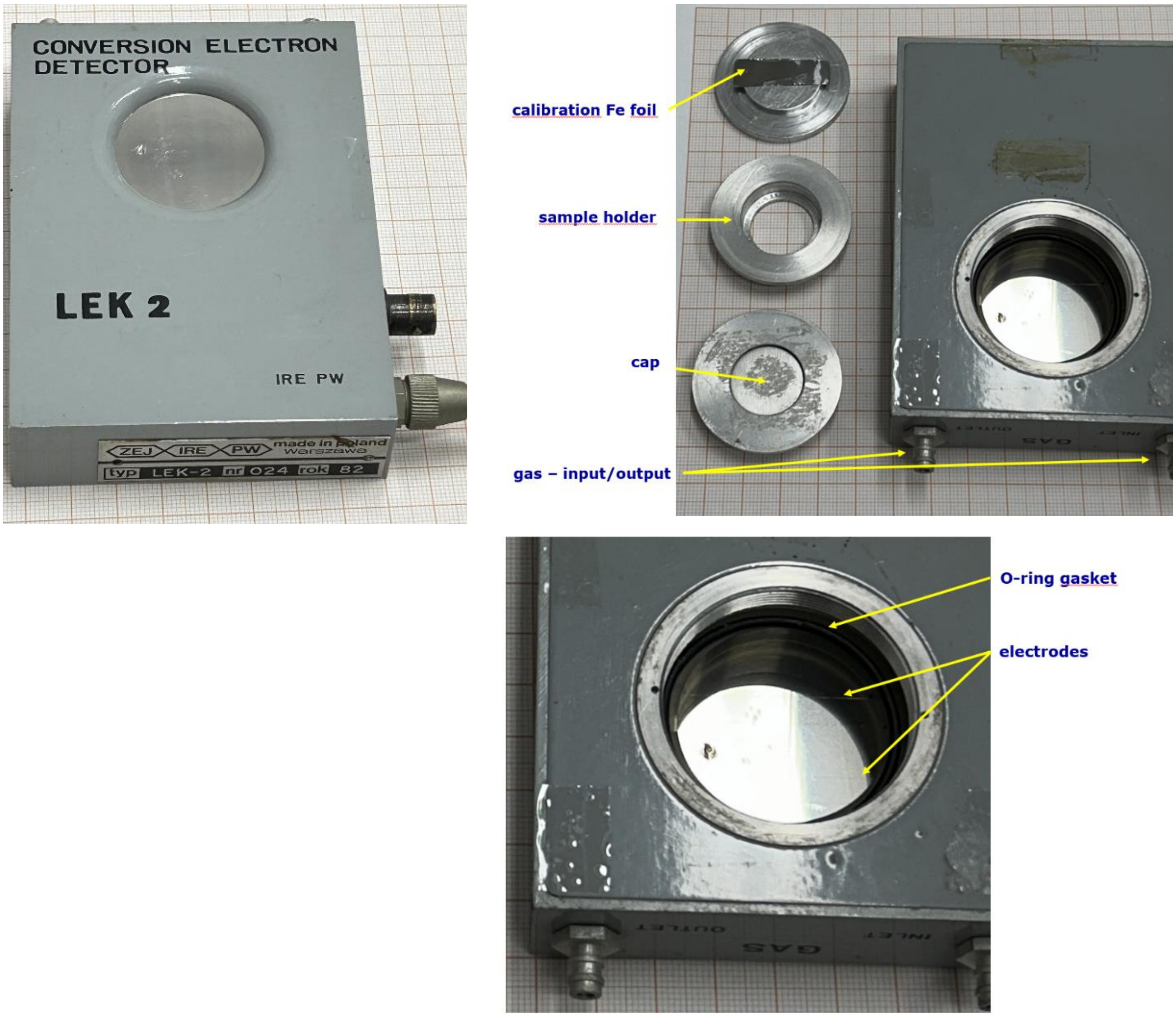


Figure 6. The LEK 2 conversion electron detector.

In this type of the counter, the sample must have certain dimensions. Area of the sample is ~ 2 – 4 cm^2 (in the form of sheet 2 cm x 2 cm or disc with diameter 2 cm). The maximum thickness of the sample is less than ~ 5 mm. The sample is mounted inside the counter. Two electrodes collect charge, which is directed to the preamplifier.

Counter MCD-14

The actual appearance of the MCD-14 detector is shown in Figures 7 and 8.

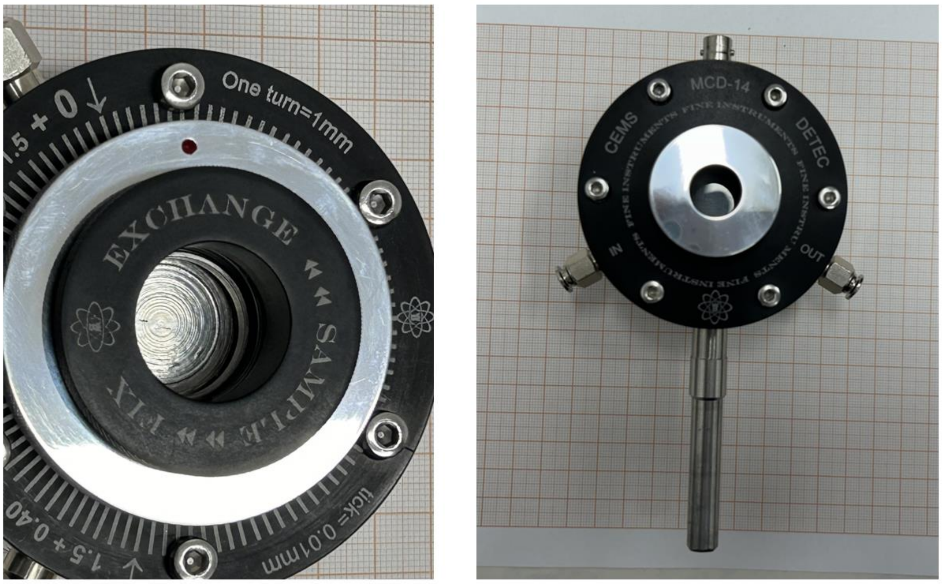


Figure 7. The MCD-14 conversion electron detector.

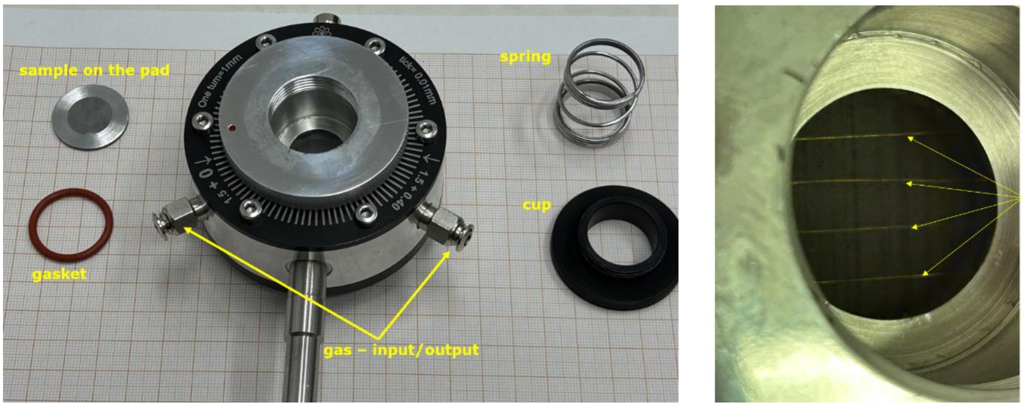


Figure 8. The elements of MCD-14 conversion electron detector and a chamber with four electrodes.

In the MCD-14 detector, the area of the sample is still ~ 2 – 4 cm^2 (in the form of sheet 2 cm x 2 cm or disc with diameter 2 cm); however, the maximum thickness of the sample is less than ~ 3 mm. The sample is mounted inside the counter and there is a possibility to change the distance between the sample and electrodes. Four electrodes collect charge, the efficiency of counting is higher than in the case of LEK 2 counter.

The real view of the mounted MCD-14 detector is presented in Figure 9.

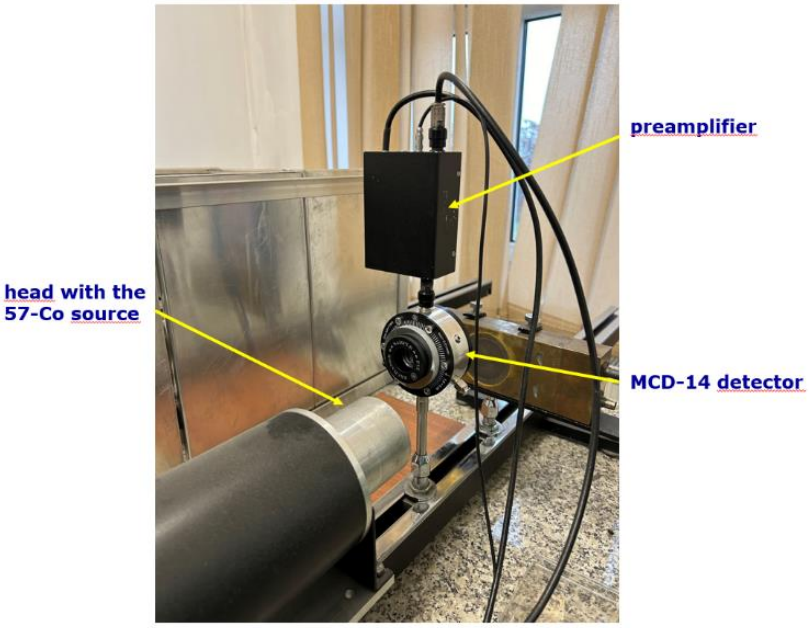


Figure 9. The experimental setup with MCD-14 counter.

5 Materials necessary for CEMS measurements

Necessary for the CEMS measurements is the radioactive source, i.e., nuclide that emits γ quanta with the suitable energy. In the case of the samples containing iron, the proper source is ^{57}Co which decays during electron capture to the ^{57}Fe stable isotope (scheme of the decay is shown in Figure 10).

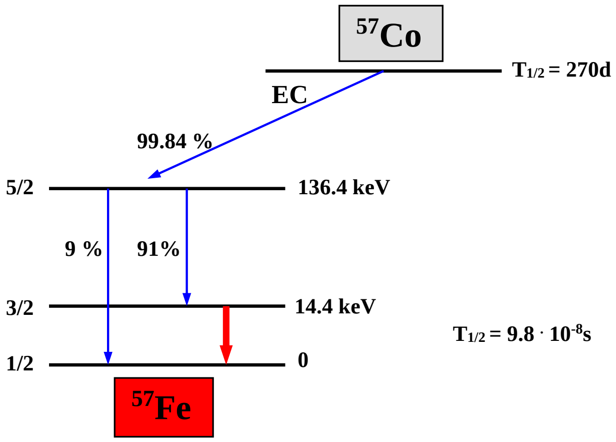


Figure 10. The scheme of the ^{57}Co radioactive decay.

In measurements, the energy of γ quanta 14.4 keV is used. The activity of the source about 1 GBq is enough to lead the effective measurements for 3 years. The sketch of the source ^{57}Co in Rh matrix is presented in Figure 11. The dimensions given are standard and fit all heads in Mössbauer spectrometers used in laboratories around the world. The minimal activity of the source necessary for measurements is about 1 GBq.

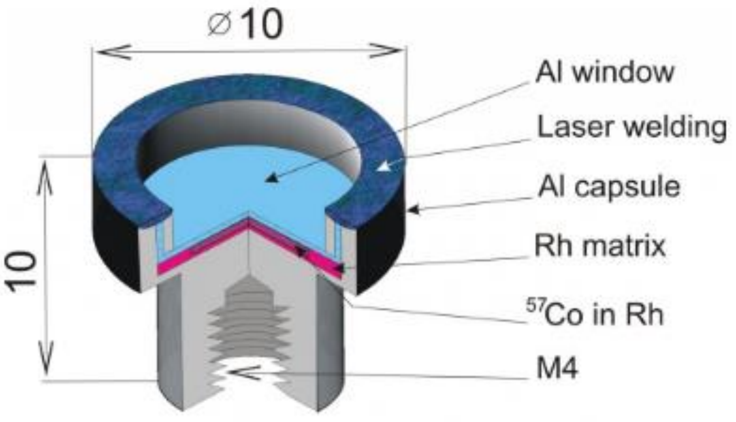


Figure 11. The sketch of the source ^{57}Co with cover and mounting element (M4).

The second important element is the mixture of gases. In the case of composition 90 % helium and 10 % methane, the supply voltage on the electron counter is typically in the range of 1000 – 1300 V. The volume of 50 liters is enough to carry out measurements by 1 year.

6 CEMS procedure

The procedure of CEMS measurements includes three main steps.

6.1 Preparing the spectrometer

To prepare the spectrometer to measurements is needed:
a) installation of a reference (calibration) sample - α-Fe foil with a thickness of 25 μm;
b) installation of an internal conversion electron detector in the spectrometer;
c) regulation of the optimal gas flow through the detector (90 % He + 10 % CH_4 mixture);
d) recording a continuous energy spectrum of secondary electrons resulting from the phenomenon of the internal conversion, observing electrical impulses on an oscilloscope;
e) selection/correction of operating parameters based on the observed pulse spectrum (electron energy spectrum):
- supply voltage of the electron counter (voltage between the sample and the electrodes), typically in the range 1000 – 1300 V;
- gain of the amplitude of electrical impulses from the counter, usually 40 – 100 times;
- pulse width 0.5 – 2 μs,
- selecting the pulse discrimination threshold - cutting off electronic noise; the discrimination threshold is usually in the range of 50 – 100 mV depending on the set gain.

6.2 Calibration of the spectrometer transducer velocity

In the case of measurements in constant acceleration mode:

a) selection of the maximum velocity (amplitude of the triangular signal) adequate to the expected spectrum of the tested sample;
b) starting the spectrometer and recording the calibration spectrum; for a radioactive source with an activity of 1 GBq collecting time is between 2 and 6 h;
c) automatic calibration – assigning a speed (energy) value to each time channel in which the pulses were recorded (it is performed automatically by the software).

6.3 Sample measurement

Main sample measurement includes:

a) replacing the calibration sample by the test sample; the sample should be conductive and have a thickness proper to the type of counter;
b) selecting the appropriate distance between the sample and the detector electrodes (according to the sample thickness);
c) starting the measurement with the spectrometer operating parameters identical to those of the calibration sample; the measurement lasts from 1 day to 2 weeks depending on the sample type and iron content;
d) recording the Mössbauer spectrum with the appropriate number of detection channels; the maximum possible number of channels is 2 x 2048; this choice compromises between a sufficiently large counting statistic and a sufficiently small speed step (energy resolution); typically, the spectrum is registered in 2 x 512 channels.

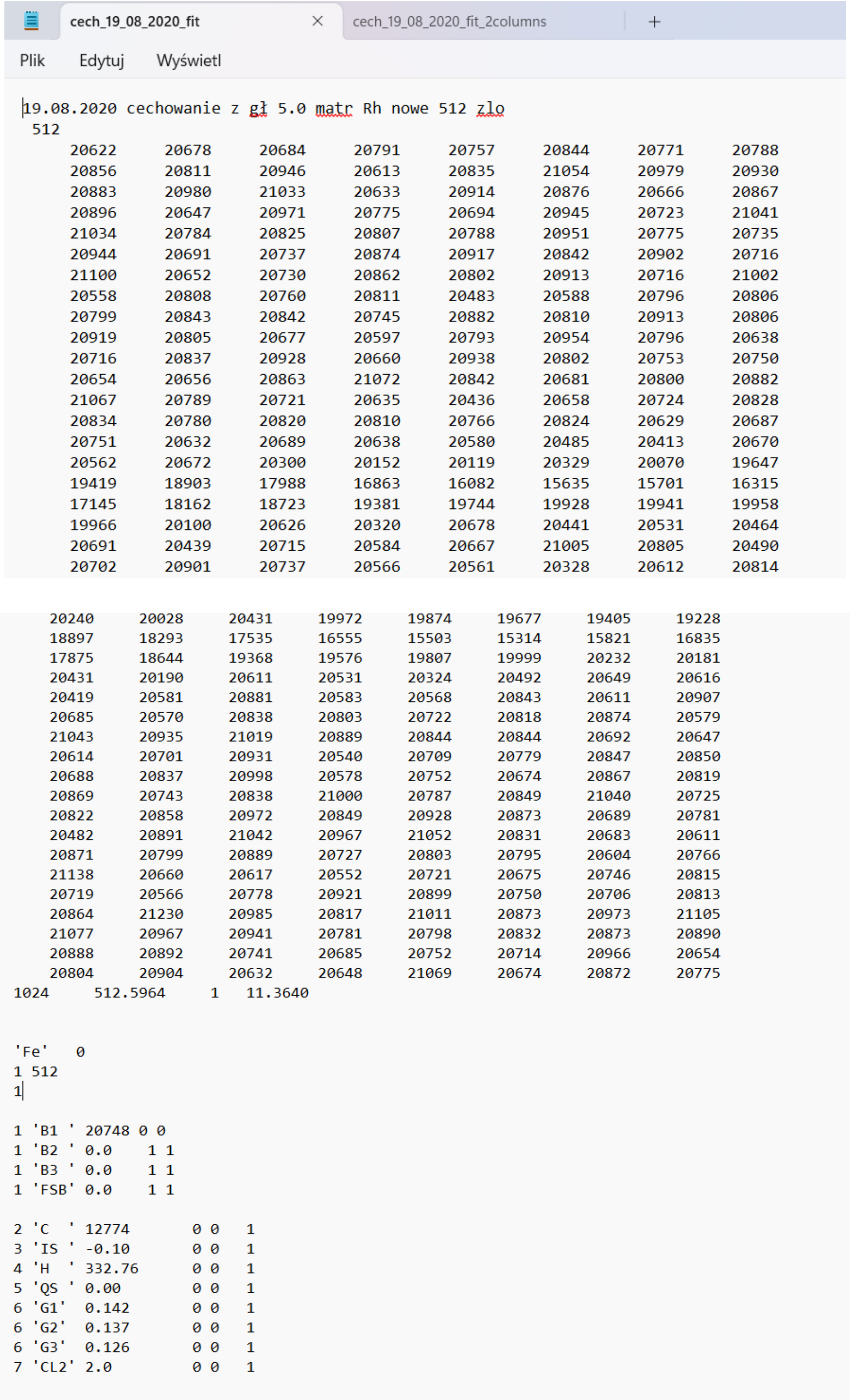
6.4 Software

The software used, program MOS, was elaborated by Jan Żukrowski on the basis of the papers [7-11]. The numerical program allows fitting single lines, doublets and Zeeman sextets depending on the kind of Mössbauer spectrum. The maximum number of the components is 36.
As a results of fitting, the following parameters are obtained for the suitable component:
- hyperfine interactions parameters – isomer shift, quadrupole splitting (shift), hyperfine magnetic field;
- spectral parameters – amplitude of lines, half width at half maximum, ratios intensity of spectral lines 1 to 3 and 2 to 3, area of spectral lines.

The Lorenzian shape of spectral lines is assumed during the fitting procedure.

The MOS software works in an iterative manner. It allows one to obtain the best set of hyperfine interactions and spectral parameters, i.e., the set giving the lowest $\chi 2$ parameter.

Experimental data can be loaded in two types of files:
a) an 8-column text file with the number of counts in each channel separated by tab (Figure 12);
b) a 2-column text file with the first column being velocity and, the second column being the counts number for each channel (Figure 13).
The starting hyperfine parameters for the fitting procedure are gathered at the end of the file as shown in Figure 12. Besides that, just below the experimental data, number of channels, folding point, and maximum velocity must be included.



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     20883     20980     21033     20633     20914     20876     20666     20867
     20896     20647     20971     20775     20694     20945     20723     21041
     21034     20784     20825     20807     20788     20951     20775     20735
     20944     20691     20737     20874     20917     20842     20902     20716
     21100     20652     20730     20862     20802     20913     20716     21002
     20558     20808     20760     20811     20483     20588     20796     20806
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     20919     20805     20677     20597     20793     20954     20796     20638
     20716     20837     20928     20660     20938     20802     20753     20750
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     21067     20789     20721     20635     20436     20658     20724     20828
     20834     20780     20820     20810     20766     20824     20629     20687
     20751     20632     20689     20638     20580     20485     20413     20670
     20562     20672     20300     20152     20119     20329     20070     19647
     19419     18903     17988     16863     16082     15635     15701     16315
     17145     18162     18723     19381     19744     19928     19941     19958
     19966     20100     20626     20320     20678     20441     20531     20464
     20691     20439     20715     20584     20667     21005     20805     20490
     20702     20901     20737     20566     20561     20328     20612     20814

     20240     20028     20431     19972     19874     19677     19405     19228
     18897     18293     17535     16555     15503     15314     15821     16835
     17875     18644     19368     19576     19807     19999     20232     20181
     20431     20190     20611     20531     20324     20492     20649     20616
     20419     20581     20881     20583     20568     20843     20611     20907
     20685     20570     20838     20803     20722     20818     20874     20579
     21043     20935     21019     20889     20844     20844     20692     20647
     20614     20701     20931     20540     20709     20779     20847     20850
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     20869     20743     20838     21000     20787     20849     21040     20725
     20822     20858     20972     20849     20928     20873     20689     20781
     20482     20891     21042     20967     21052     20831     20683     20611
     20871     20799     20889     20727     20803     20795     20604     20766
     21138     20660     20617     20552     20721     20675     20746     20815
     20719     20566     20778     20921     20899     20750     20706     20813
     20864     21230     20985     20817     21011     20873     20973     21105
     21077     20967     20941     20781     20798     20832     20873     20890
     20888     20892     20741     20685     20752     20714     20966     20654
     20804     20904     20632     20648     21069     20674     20872     20775
1024      512.5964     1   11.3640


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1 512
1

1 'B1 ' 20748 0 0
1 'B2 ' 0.0    1 1
1 'B3 ' 0.0    1 1
1 'FSB' 0.0    1 1

2 'C  ' 12774      0 0   1
3 'IS ' -0.10      0 0   1
4 'H  ' 332.76     0 0   1
5 'QS ' 0.00       0 0   1
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6 'G2'  0.137      0 0   1
6 'G3'  0.126      0 0   1
7 'CL2' 2.0        0 0   1
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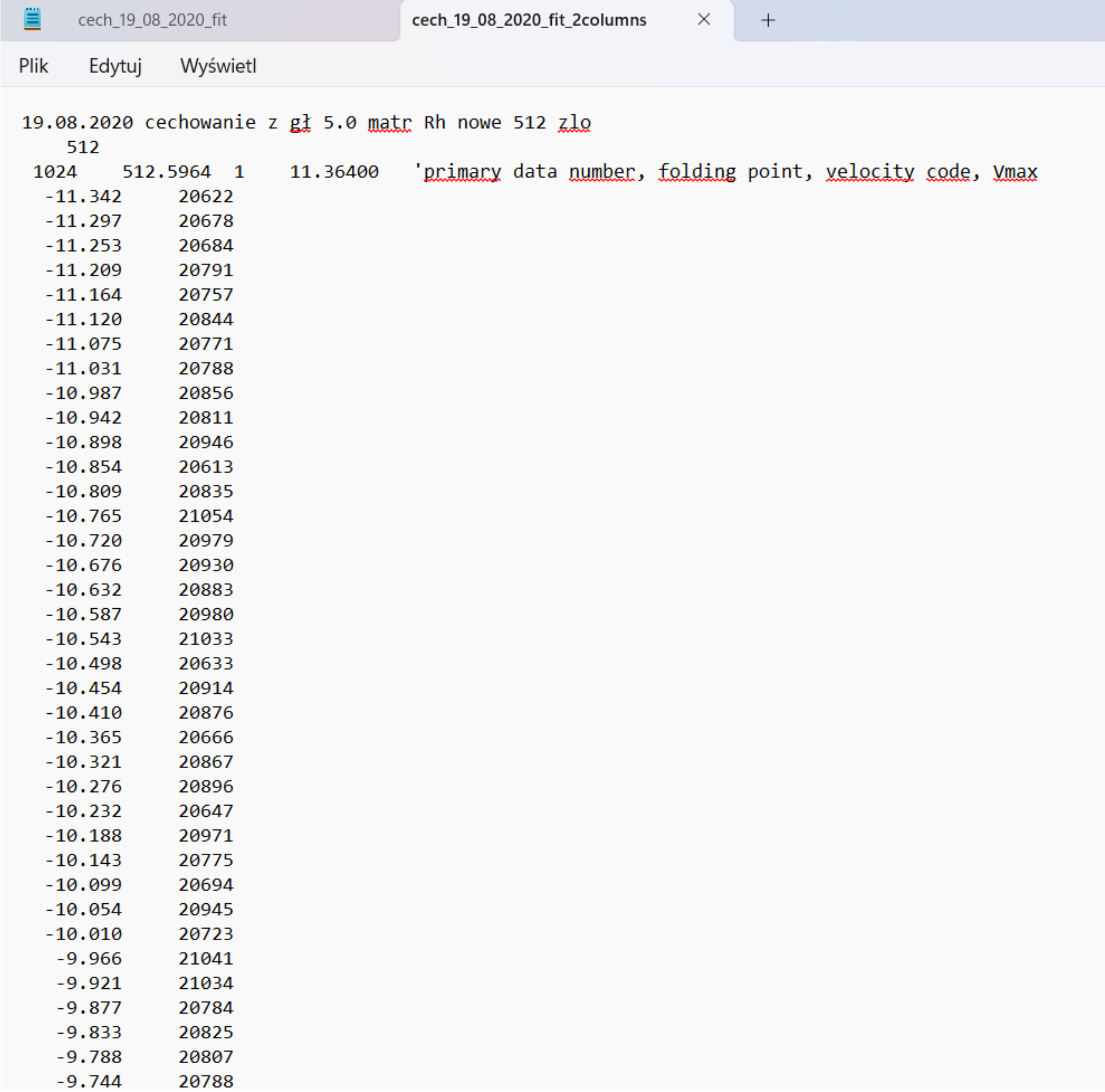
Figure 12. The input file with experimental data and starting hyperfine parameters in the 8-column text file.



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19.08.2020 cechowanie z gł 5.0 matr Rh nowe 512 zlo
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 1024     512.5964  1     11.36400    'primary data number, folding point, velocity code, Vmax
  -11.342     20622
  -11.297     20678
  -11.253     20684
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  -11.075     20771
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  -10.809     20835
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   -9.921     21034
   -9.877     20784
   -9.833     20825
   -9.788     20807
   -9.744     20788
```

Figure 13. Experimental data in the 2-column text file.

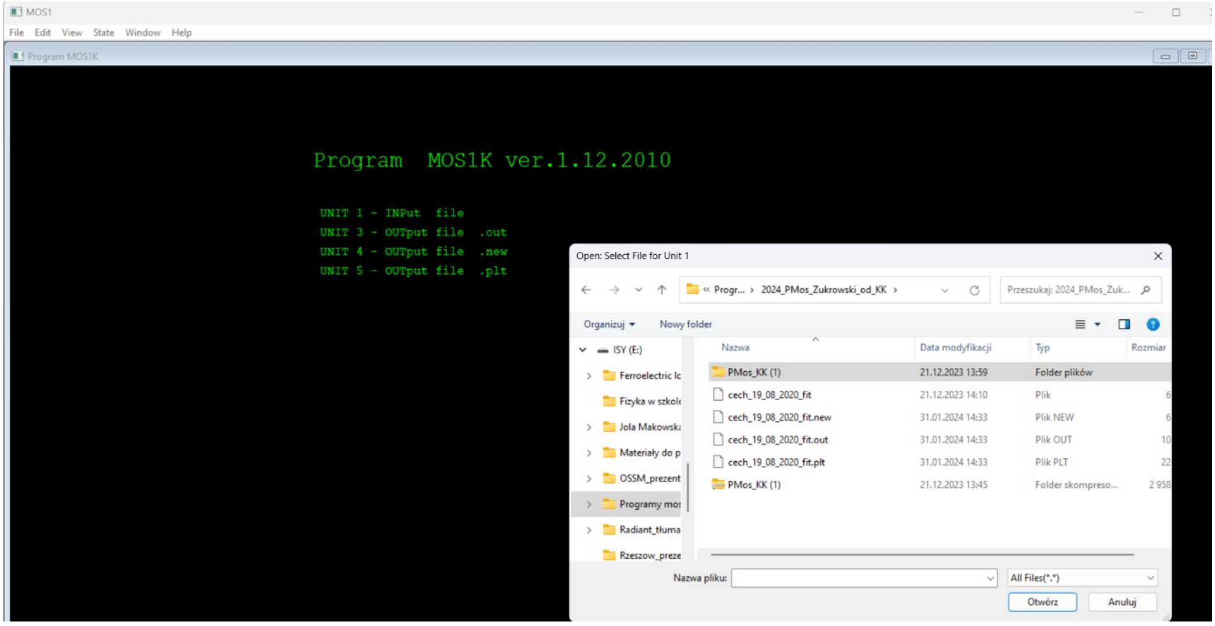
The latest version of the MOS software is MosK program. The advantage of the MosK software is the possibility of pre-processing of experimental data (Figure 14). Among the most useful operations possible in this step are the following:
- folding of the unfolded spectrum, either in automatic or manual mode;
- change of number of channels for the registered spectrum. This step allows one to choose the best channel number, i.e., the number that gives the best signal-to-noise ratio with acceptable energetic resolution;
- adding two spectra in the case of 2-spectra were being registered simultaneously, e.g., by use of 14.4 keV line and the “escape peak”;
- including calibration data;
- closer inspection of the spectrum in the “point-by-point” manner.

V(t) = lin.
Effect = 26.22 %
Ymax = 21230
Vmax = 11.364 mm/s
Fx = 512.60 chan.
Ymin = 15314

19.08.2020 cechowanie z gł 5.0 matr Rh nowe 512 zlo

Figure 14. The front panel of the MosK software.

Loading of the input file is executed by clicking file ->OpenSourceFile (Figure 15).



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Program  MOS1K ver.1.12.2010

 UNIT 1 - INPut  file
 UNIT 3 - OUTput file  .out
 UNIT 4 - OUTput file  .new
 UNIT 5 - OUTput file  .plt
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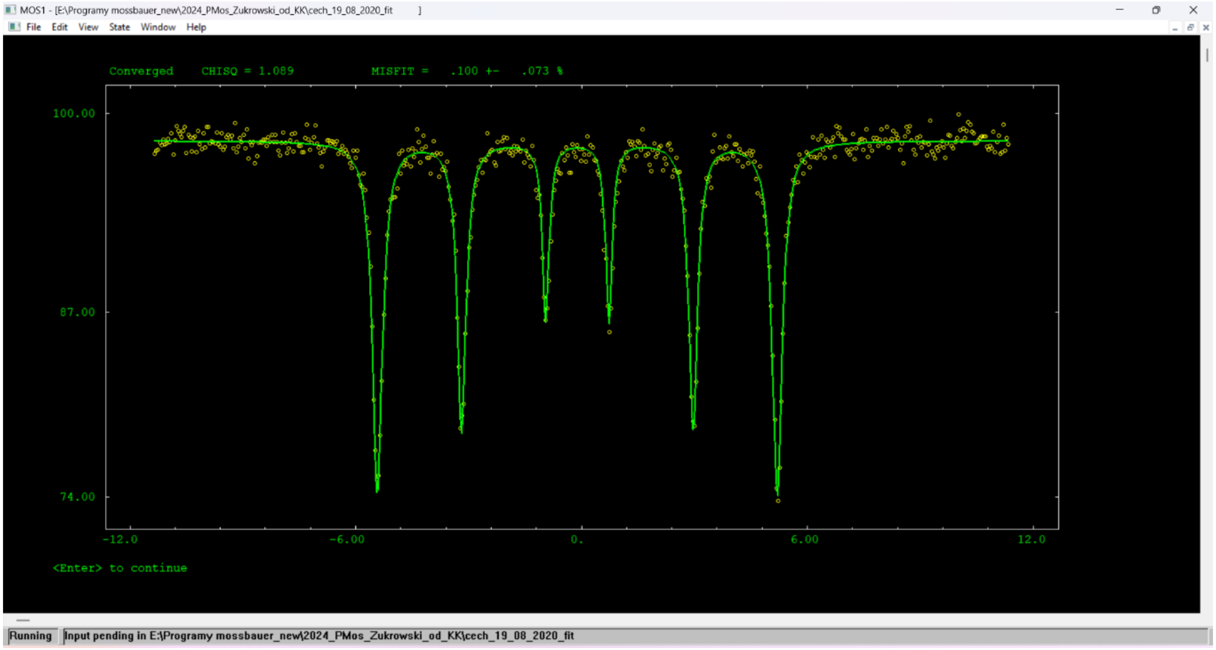
PMos_KK (1)	21.12.2023 13:59	Folder plików	
cech_19_08_2020_fit	21.12.2023 14:10	Plik	6
cech_19_08_2020_fit.new	31.01.2024 14:33	Plik NEW	6
cech_19_08_2020_fit.out	31.01.2024 14:33	Plik OUT	10
cech_19_08_2020_fit.plt	31.01.2024 14:33	Plik PLT	22
PMos_KK (1)	21.12.2023 13:45	Folder skompreso...	2 958

Figure 15. The page for Source File loading in the MosK ver. 12.2010 software.

After loading the source file, the window with the experimental and calculated pattern is opened (Figure 16). The last step is an iterative spectrum fitting using the internal procedure based on the χ2 minimization. After this step, the user must check whether the procedure was convergent. If not, the last step must be repeated with the starting hyperfine interactions parameters changed. After fitting is done, the software produces output text file with the data ready for importing in a graphical software like excell, origin, grapher etc..



```
Converged    CHISQ = 1.089          MISFIT =   .100 +-   .073 %
```

```
<Enter> to continue
```

```
Input pending in E:\Programy mossbauer_new\2024_PMos_Zukrowski_od_KK\cech_19_08_2020_fit
```

Figure 16. The window of the MosK software with an experimental and calculated Mössbauer spectrum.

7 Results of measurements

The calibration of the spectrometer should be performed twice, i.e., before and after the measurement of the given sample. From these two measurements, the average value of the maximum velocity (in the last channel) is determined. This value is necessary for applying in the numerical procedure of the Mössbauer spectrum elaboration. The calibration spectrum (taken for α-iron) is presented in Figure 17.

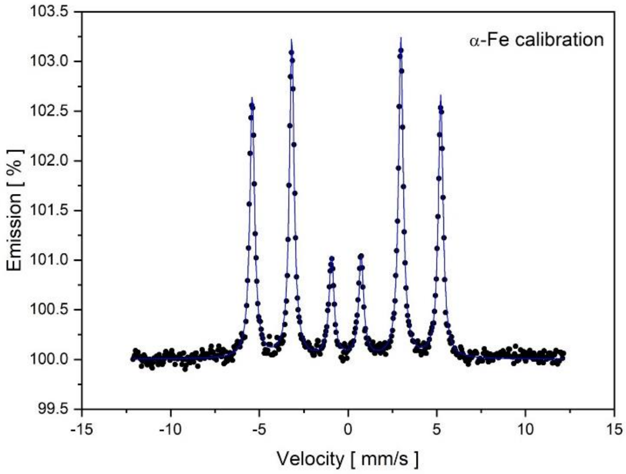


Figure 17. Room-temperature calibration CEMS spectrum on α-iron foil.

From the numerical fit of the spectrum, the hyperfine interactions parameters are obtained. There are:
- isomer shift *IS* = 0.01(1) mm/s (the value is relative to α-iron and should be zero);
- hyperfine magnetic field B_{hf} = 33.06(2) T;
- quadrupole shift *QS* = -0.003(5) mm/s (the value should be zero because iron has cubic lattice).

All the values are characteristic of α-iron.
The half width of the spectral lines Γ = 0.16(2) mm/s is near the natural half width.

After calibration, the measurement for the proper sample is performed. In Figure 18, the CEMS spectrum for the steel sample Fe with 2.9 wt.% of Si is presented as an example.

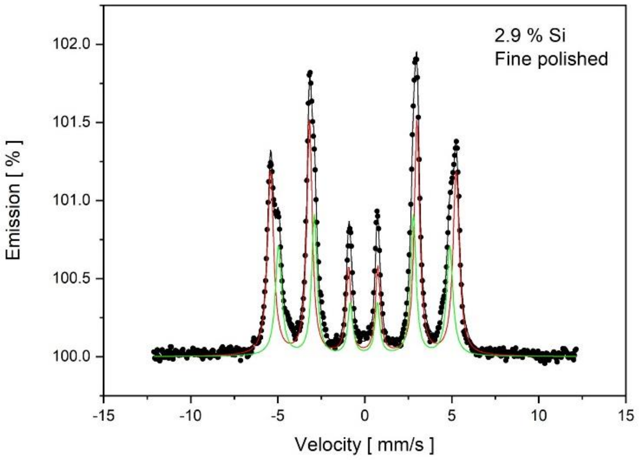


Figure 18. Room-temperature CEMS spectrum of the sample Fe-2.9 wt.% Si with two components marked by red and green lines.

The main goal of the elaboration of the Mössbauer spectrum is to determine the number of the components and explain their origination. The fit is concerned as good when all the components are physically justified and the fit parameter χ2 is near 1.

8 References

[1] M. Przybylski, *From Three-to Two-Dimensional Magnetism of Fe*, habilitation dissertation, ed. by Wydawnictwa AGH, Kraków 1997.

[2] M. Seberini, J. Sitek, *Surface Analysis of Iron-Containing samples by CEMS. An Overview of the Method.* Journal of electrical Engineering, 59 (5) 2008, 234-240.

[3] B.V. Thosar, J.K. Srivastava, P.K. Iyengar, S.C. Bhargava, *Advances in Mössbauer Spectroscopy. Applications to Physics, Chemistry and Biology.* Elsevier Scientific Publishing Company, Amsterdam-Oxford-New York 1983.

[4] Y. Garcia, J. Wang, T. Zhang, *Mössbauer Spectroscopy. Applications in Chemistry and Materials Science.* Wiley-VCH 2024.

[5] I. Bibicu, *Mössbauer Spectroscopy Applied to Surfaces Study*. Rev. Roum. Chim. 57(9-10) 2012, 877-889.

[6] W.R. Leo, *Techniques for Nuclear and Particle Physics Experiments*, 2nd ed. Springer-Verlag, Berlin, 1994.

[7] M.F. Bent, B.I. Persson and D.G. Agresti, Computer Phys. Comm. 1, 1969, 67.

[8] B.L. Christman and T.A. Tumolillo, Computer Phys. Comm. 2, 1971, 322.

[9] W. Wilson and L.J. Swartzendruber, Computer Phys. Comm. 7, 1974, 151.

[10] K. Kulcsar, D.L. Nagy and L. Pocs, Proc. Conf., Dresden, 1971, 594.

[11] S.L. Ruby, *Mossbauer Effect Methodology*, vol. 8, ed. I.J. Gruverman and C.W. Seidel, Plenum Press, New York-London 1973.