Setup of Mössbauer Spectrometers at RCPTM

Regional Centre of Advanced Technologies and Materials (RCPTM), Palacky University, Olomouc, Czech Republic

Setup of Mössbauer spectrometers (MS) for structural, phase, and magnetic characterization of iron- or tin-containing samples is presented. This comprehensive line of ^{57}Fe and ^{119}Sn Mössbauer spectrometers covers transmission spectrometers (TMS) for room temperature (RT) measurements, temperature dependent measurements and measurements in an external magnetic field. An RT Conversion Electron/Conversion X-ray Mössbauer technique (CEMS/CXMS) is also available. The main concept of the RT MS is a table-top spectrometric bench with a control unit based on special-purpose hardware or standard PC platform. The first way offers a compact design and PC independent spectra collection system. The second setup, a PC-based system, which uses commercial devices and LabVIEW software, offers easy customization and enables advancement in spectrometer construction. The both types of control systems are able to operate special parts (velocity transducers, gamma-ray detectors) of unusual spectrometric benches. The standard velocity axis range is up to ±20 mm/s with a maximum nonlinearity of 0.1%. Applicable measuring conditions of presented TMSs cover a cryogenic temperature range from 1.5 up to 300 K and high temperature range from RT up to 1000 °C. With in-field low-temperature MS, we are able to analyze samples normally in the external magnetic fields up to 8 T (in temperature interval from 1.5 up to 300 K). In addition, special modes of measurements can be applied including backscattering gamma-ray geometry or measurement in an inert or controlled-humidity atmosphere. Technical details and construction aspects of spectrometers are presented.

Introduction

Laboratory of Mössbauer spectroscopy at Regional Centre of Advanced Technology (RCPTM) is equipped with several Mössbauer spectrometers. A setup of seven MSs is daily used as a comprehensive line of ^{57}Fe and ^{119}Sn Mössbauer spectrometers which also covers temperature dependent measurements and measurements in an external magnetic field.

Main concept of the RT TMS exploits a table-top spectrometric bench with a control unit based on special-purpose hardware or standard PC platform. First way offers a compact design and PC independent spectra collection system [1]. This design can be termed as HW-based TMS or MS96 (revised at 1996). The second setup, a PC-based system, uses commercial devices and control application fully developed in LabVIEW software [2]. This design can be termed as SW-based MS or VI (Virtual Instrument).

Currently, three MS96 TMSs are used in parallel with three VI TMSs based on USB (multifunction card and high-rate digitizer) or PCI (function generator and high-rate digitizer) devices. Both types of control systems are able to operate special parts (velocity transducers, gamma-ray detectors) of unusual spectrometric benches.

Moreover, there is one spectrometer with an RT CEMS/CXMS/TMS measurement capability. This model denoted as CEMS2012 is based on continuous gas flow counter. Its construction utilizes both computer based PXI system as well as special-purpose hardware.

All spectrometers are able to utilize both common isotopes, i.e., ^{57}Fe and ^{119}Sn, in the standard velocity axis range up to ±20 mm/s with a maximum nonlinearity of 0.10%.

RT MSs are commercially available in the form of complete systems as well as their separate parts!

Transmission Mössbauer Spectrometers

Below, a line of TMSs including measurements in a broad temperature interval and in an external magnetic field is presented.

RT Mössbauer Spectrometers

The design of this RT spectrometer offers compact framework, having small dimensions and mass (420×180×150 mm in dimensions, 9 kg in mass), which can be driven by both types of control system. Compact design of RT Mössbauer spectrometer MS96 is depicted in Fig. 1.

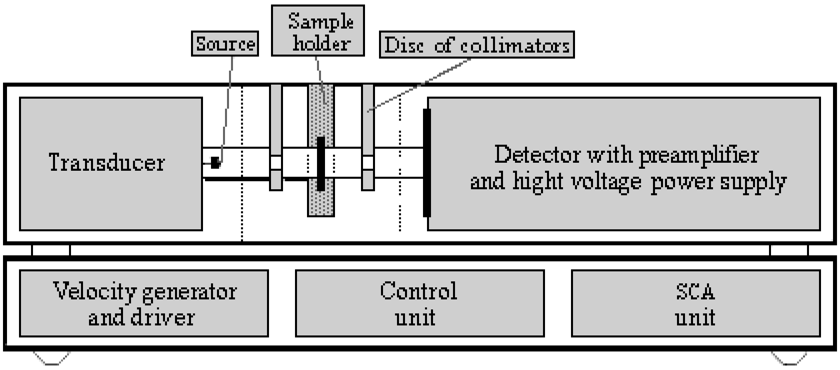

FIGURE 1. Schematic drawing of RT Mössbauer spectrometer MS96.

In all RT spectrometric benches, standard double-loudspeaker-type "mini" velocity transducers [3] and fast scintillation detectors (a photomultiplier with a YAP scintillation crystal) [4] are used.

In this configuration, an average nonlinearity of the velocity scale for all RT spectrometers which use "mini" transducer is 0.02% and the maximum nonlinearity reaches 0.05% (measured with an α-Fe standard foil in ±13 mm/s velocity interval).

Real picture of the spectrometric bench is shown in Fig. 2. Mössbauer spectrometers based on VI concept use the same bench (transducer, collimators, sample holder, and detector); however, lower units (velocity, control, and SCA) are not implemented as they are replaced by mentioned commercial available devices with a personal computer.

In addition, special modes such as backscattering mode and measurements in an inert atmosphere are also available depending on the issue studied. For these purposes, one of TMS is kept all the time in the glove box filled with nitrogen atmosphere.

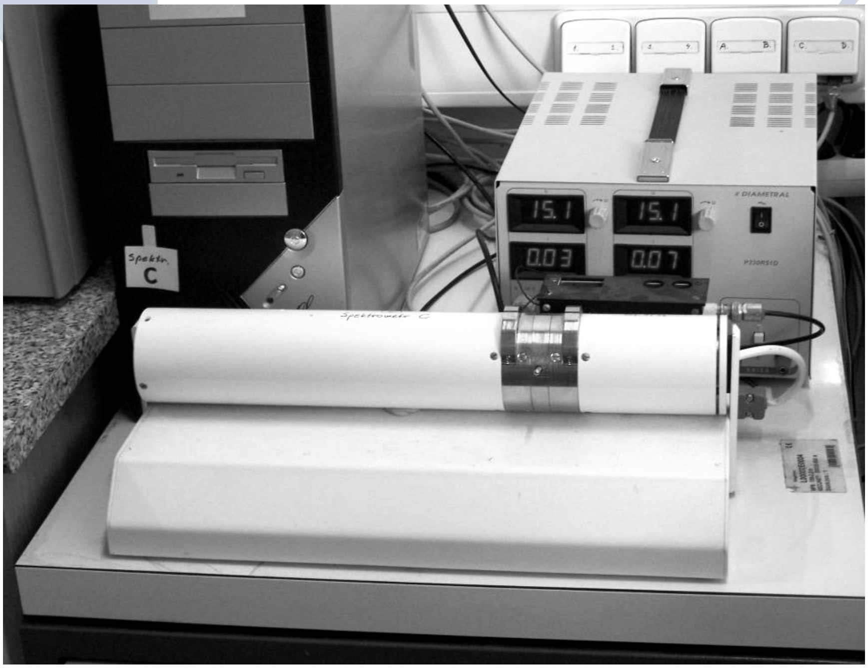

FIGURE 2. Picture of RT TMS.

Low-Temperature Mössbauer Spectrometer

A standard low-temperature MS consists of closed-cycle helium refrigerator equipped with the Mössbauer spectrometer. A CCS-850 cryostat of Janis model is designed to operate at temperatures from 12 to 300 K. The system is equipped with a vibration isolation bellows and mounting flange assembly. This assembly is designed to absorb vibrations generated by the refrigerator.

In-Field Low-Temperature Mössbauer Spectrometer

This Mössbauer spectrometer utilizes Spectromag system (Oxford Instruments). Spectromag is a superconducting split-pair, horizontal field magnet system and provides an access to a sample in a variable magnetic field/low temperature environment.

With an in-field low-temperature (IFLT) MS, it is possible to analyze samples normally in the external magnetic fields (parallel orientation of the external magnetic field with respect to the gamma-rays propagation) up to 8 T (in the temperature interval from 1.5 up to 300 K). Moreover, this spectrometer is able to record spectra in a wide velocity axis range up to ±55 mm/s.

A velocity driving system of this spectrometer uses a more robust design of the velocity transducer with a digital PID controller [5,6]. Real picture of the IFLT MS is shown in Fig. 3.

In this configuration, an average nonlinearity of the velocity scale is 0.10% and the maximum nonlinearity reaches 0.16% (measured with an α-Fe standard foil in ±13 mm/s velocity interval).

FIGURE 3. Picture of the IFLT MS.

High-Temperature Mössbauer Spectrometer

In this spectrometer, a common RT TMS is modified – a sample is placed into the furnace instead of the holder. Other parts of the spectrometer are identical as in the case of RT TMS (see Fig. 4 and 5).

Available temperature ranges from RT up to 1000 °C with inert atmosphere (nitrogen) possibility. Depending on requirements, spectra are possible to collect at a given time period for a desired time interval, thus being suitable for studying fast transformation processes (involving oxidation, humidity, etc.) [7].

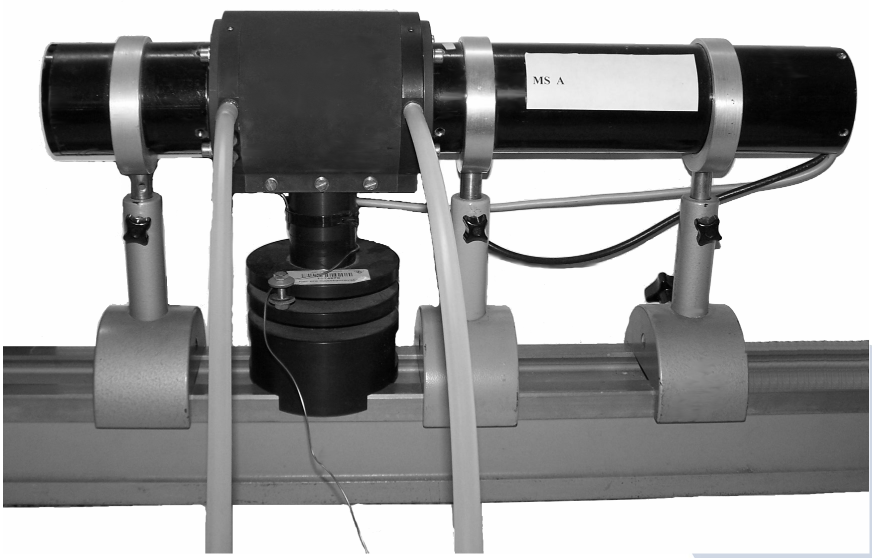

FIGURE 4. Spectrometric bench for high temperature MS with furnace used instead of the sample holder.

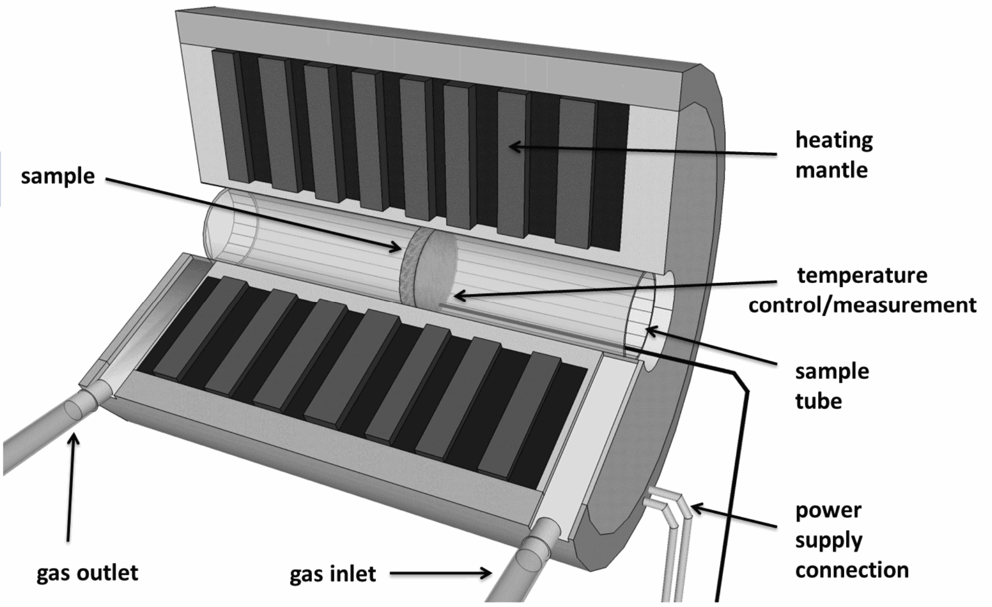

FIGURE 5. Schematic view of the furnace with sample position depicted. Sample is placed in the quartz glass tube between the quartz wool as a sandwich.

CEMS/CXMS/TMS Spectrometer

RCPTM Mössbauer laboratory also possesses and provides RT CEMS/CXMS/TMS spectrometer (see Fig. 6) denoted as CEMS2012, which is based on a VI concept and industrial PXI system (devices work as a function generator and high-rate digitizer) [8]. The apparatus is equipped with a proportional continuous gas flow counter for accumulation of integral CEMS or CXMS spectra and with a scintillation counter for simultaneous TMS recording. A Penning mixture of He+10% CH_4 and Ar+10% CH_4 serves as a counting gas for CEMS and CXMS, respectively.

The spectrometer design was revisited during the year 2012. Partial improvements include a more efficient pre-amplifier and redesigned CEMS/CXMS counter. New design of the counter includes a multiwire anode holder with a more accurate geometry, a new collimation system with adjustable sample-to-source distance, and Kapton windows with a precise O-ring sealing. It has a beneficial impact on higher counting rates or shorter counting times.

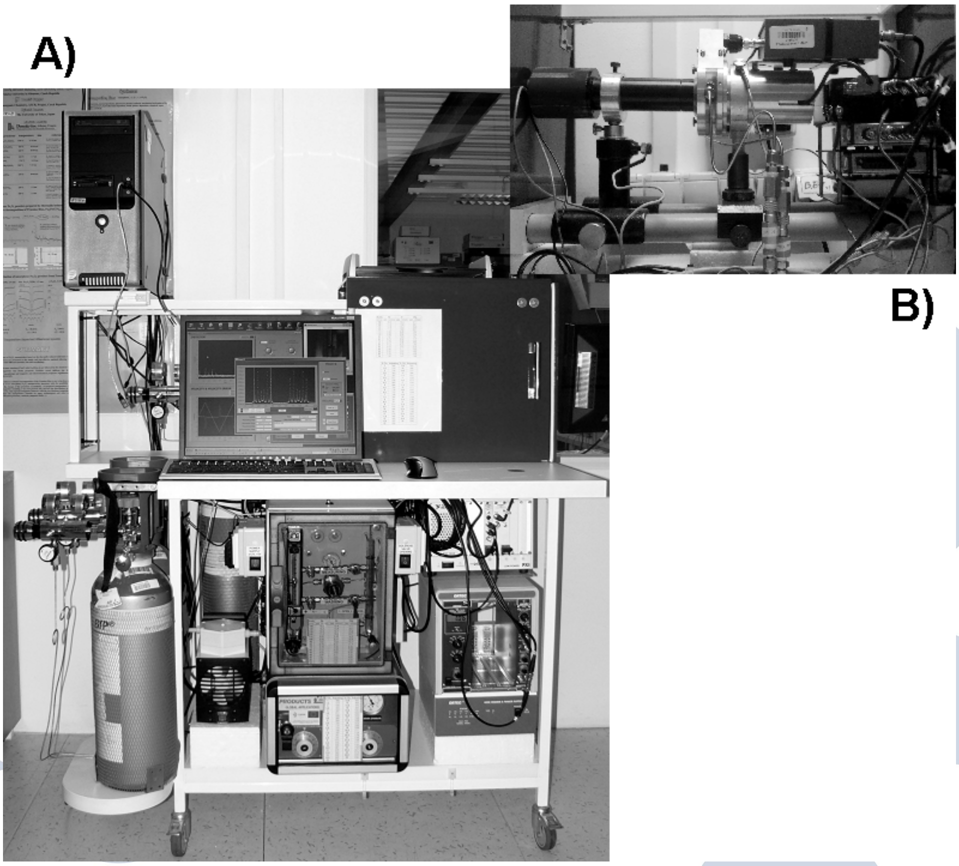

FIGURE 6. (a) CEMS2012 spectrometer and (b) detail of opened shielded chamber with a TMS detector on the left side of the chamber and CEMS/CXMS counter in the middle.

Supplements

One of VI TMS is used for "experimental" work, where new methods and techniques for spectrometric parts development and data manipulation are applied and evaluated.

Currently, a new design of electronic units for Mössbauer spectrometric bench is under construction. The goal of this project is to develop Mössbauer spectrometer, which is will meet the requirements of up-to-date laboratory equipment. New parts will be constructed as special-purpose devices with embedded features for test and control functions (more autonomous units with self-diagnostic tasks, computer server, data storage, backup devices, etc.).

Another project, having already started in the laboratory, deals with a time-differential Mössbauer spectrometer (TDMS) in which the Mössbauer effect of the 14.4 keV transition as well as the time sequence of the 14.4–122 keV γ-γ cascade following the electron capture of ^{57}Co is registered. TDMS measurements, however, have a number of experimental difficulties to be handled such as long measuring times and requirement of simultaneous good energy and time resolution. Virtual instrumentation technique with

LabVIEW was chosen for this purpose since it is powerful tool for solving different types of tasks implemented in TDMS.

The last planned project which we would like to mention here focuses on adaptation of a CEMS/CXMS/TMS spectrometer for Mössbauer measurements at cryogenic temperatures between RT and liquid nitrogen (LN_2) temperature. The first step to achieve this is to develop a universal system for automated LN_2 filling (see Fig. 7).

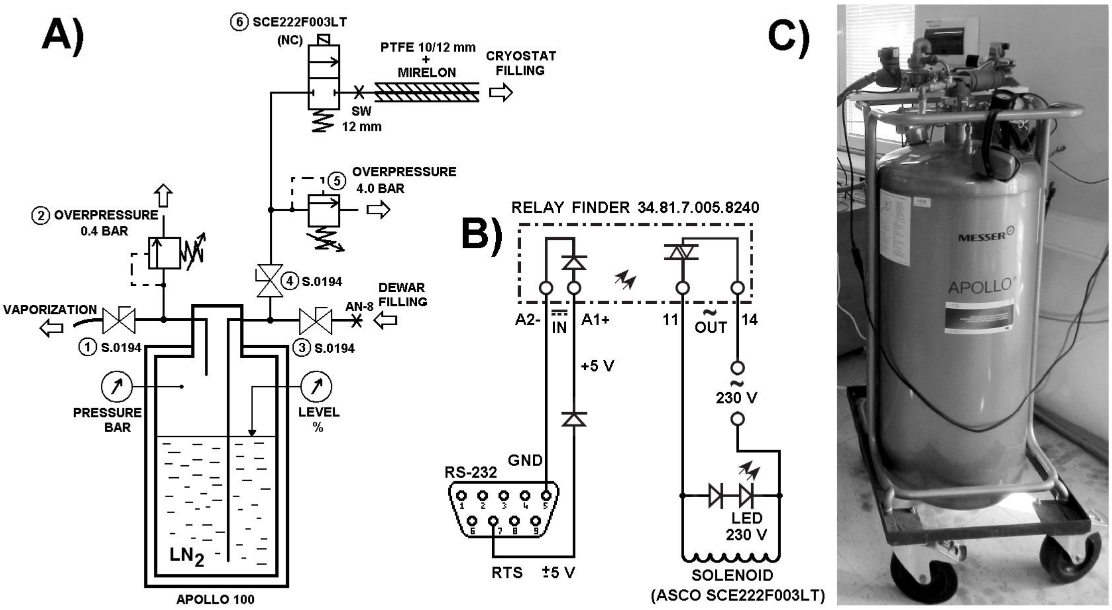

FIGURE 7. System for automated LN_2 filling: (a) valve scheme, (b) electric scheme, and (c) real view.

References

1. M. Mashlan, D. Jancik, D. Zak, F. Dufka, V. Snasel, and A. L. Kholmetskii: in Mössbauer Spectroscopy in Materials Science (Eds. M. Miglierini and D. Petridis). Kluwer Academic Publishers, Dordrecht, 1999, p. 399–406.
2. J. Pechousek, R. Prochazka, D. Jancik, M. Mashlan and J. Frydrych, *J. Phys. Conf. Ser.* **217**, 012006 (2010).
3. V. A. Evdokimov, M. Mashan, D. Zak, A. A. Fyodorov, A. L. Kholmetskii and O. V. Misevich, *Nucl. Instrum. Meth. B* **95**, 278–280 (1995).
4. M. Mashlan, D. Jancik, A. Kholmetskii, A. Mulaba and H. Pollak, *Hyperfine Interact.* **120/121**, 769–773 (1999).
5. J. Pechousek, R. Prochazka, M. Mashlan, D. Jancik and J. Frydrych, *Meas. Sci. Technol.* **20**, 017001 (2009).
6. J. Pechousek, D. Jancik, V. Evdokimov and R. Prochazka, *Nucl. Instrum. Meth. B* **267**, 846–848 (2009).
7. L. Machala, R. Zboril, V.K. Sharma and Z. Homonay, in Mössbauer Spectroscopy in Materials Science (Eds. M. Mashlan and R. Zboril). AIP Conf. Proc. 1070, New York, 2008, p. 114–121.
8. J. Frydrych, M. Mashlan, R. Zboril, J. Pechousek and M. Hermanek, *Acta Met. Slovaca* **13**, 298–301 (2007).

Contact for technical and business communication: RNDr. Jiří Pechoušek, Ph.D.,
@ jiri.pechousek@upol.cz
✉ Šlechtitelů 11, 78371 Olomouc
☎ 58 563 4949

Contact for business communication: Doc. RNDr. Libor Machala, Ph.D.,
@ libor.machala@upol.cz
✉ Šlechtitelů 11, 78371 Olomouc
☎ 58 563 4959