

Development of system and technology for mössbauer spectroscopic microscope

Kazuo Hayakawa · Yuki Akiyama · Yoshinori Tsukamoto · Mikio Kurata · Kenichi Yukihira · Hiroyoshi Soejima · Yutaka Yoshida

© Springer Science+Business Media B.V. 2011

Abstract We have been developing a “Mössbauer Spectroscopic Microscope (MSM)” which consists of a focusing lens for 14.4 keV γ -rays and a high precision X–Y stage. The measuring system both for electrons and γ -rays combined with a new Mössbauer driver, i.e., “a moving coil actuator with a liner encoder” enables us to measure the mapping images simultaneously corresponding to different spectral components. The system has a controlling system based on a LabVIEW program and a LIST mode data acquisition system (NIKI-GLASS/A3100). To investigate a correlation between the microstructure of a sample and ^{57}Fe atoms, a scanning electron microscope (APCO/Mini-EOC) is also installed to this system.

Keywords Mapping · Focusing γ -rays · Multi-capitally lens (MCX) · Fresnel zone plate (FZP) · LIST-mode data acquisition · Mössbauer spectroscopic microscope

1 Introduction

Material properties are known to depend strongly on the microstructures. A Mössbauer spectrum provides atomistic information at the nuclear probes through hyperfine interactions, but it gives only an average within an area irradiated by the γ -rays, i.e., typically 10 mm ϕ in diameter. In order to control the fine structures of state-of-the-art microelectronic devices as well as steel products down to several 10 nm, there exist a strong interest in a mapping technique for bulk materials, providing the space resolution from 1 mm down to 10 nm. In the case of ^{57}Fe Mössbauer

K. Hayakawa · Y. Akiyama · Y. Tsukamoto · M. Kurata · K. Yukihira · Y. Yoshida (✉)
Shizuoka Institute of Science and Technology, Toyosawa 2200-2, Fukuroi-city,
Shizuoka 437-8555, Japan
e-mail: yoshida@ms.sist.ac.jp

H. Soejima
Shimadzu Corporation, Nishinokyo-Kuwabaracho 1, Nakagyō-ku, Kyoto 604-8511, Japan

Fig. 1 Main panel of the LabVIEW operating system for Mössbauer spectroscopic microscopy



spectroscopy, we have recently developed a microscopy [1–3] using a focusing lens for 14.4 keV- γ -rays, i.e., multi-capillary X-ray lens (MCX) [4]. The microscopy provides a space resolution of $200\mu\text{m}$ so far, and we are further challenging to achieve the resolution down to sub-micrometer range using a combination lens of MCX with a Fresnel Zone Plate (FZP) [3]. In this paper, we report mainly on: (1) a user operation system using LabVIEW program, (2) a LIST-mode data accumulation system and a controlling system of X–Y stage, (3) a new Mössbauer driver suitable for a constant velocity operation.

(1.) User operation system using LabVIEW program:

Figure 1 shows the main panel of the LabVIEW operating system developed on Windows 7-PC for the Mössbauer Spectroscopic Microscopy (MSM). The main panel contains several sub-panel buttons corresponding to the different operations, i.e., CCD/SEM operations, setting experimental parameters, setting mapping area, mapping measurement, displaying the results. In a typical operation, we firstly start observing a sample either by a CCD camera or by a Scanning Electron Microscopy (SEM, APCO/Mini-EOC). Secondary, we define the mapping area and the magnification by clicking the mouse on the display, yielding the control parameters for an X–Y stage controller which is connected to the main PC via RS232C. The accumulation time at each mapping point and the number of mapping step, which are typically 30 s and 80×80 points, respectively, will be given from the key board.

(2.) Measuring system

The measuring system consists of a LIST-mode data acquisition system (NIKI GLASS/A3100), a three-stage micro-channel-plate (MCP/HAMAMATSU) with a center hole for conversion and Auger electrons, a Si-PIN detector (AMP TEK) for 14.4 keV γ -rays, and the related standard modules (ORTEC) for the detector signals. The sample holder is fixed on an X–Y stage mounted in a vacuum chamber. The whole system configuration is shown in Fig. 2. A special program has been developed for the Mössbauer Spectroscopic Microscopy (MSM) to convert the three sets of the LIST-mode data as functions of time, into three sets of MCS data, i.e., a standard Mössbauer data as function of the Doppler velocity. These MCS data are read by the main LabVIEW program, and subsequently, the mapping data are produced by

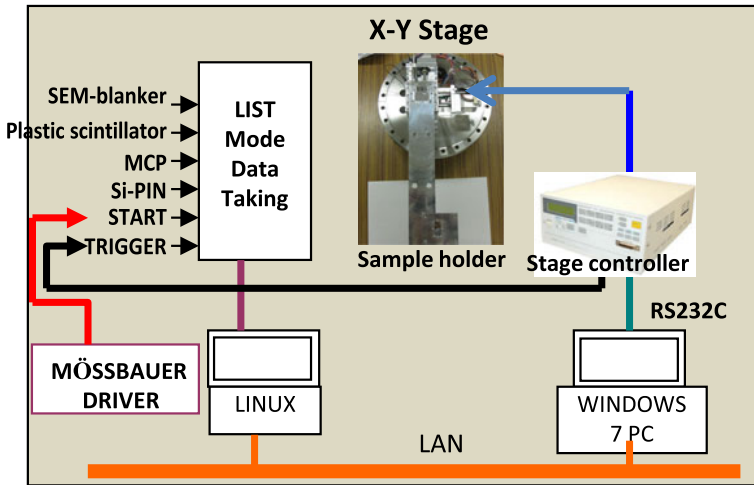


Fig. 2 System configuration: LIST-mode DATA taking system on LINUX PC and LabVIEW program on Windows PC, both of which are connected to X-Y stage controller and Mössbauer driver

summing up all data from a certain velocity range and are finally plotted as function of the position of the focused γ -ray spot.

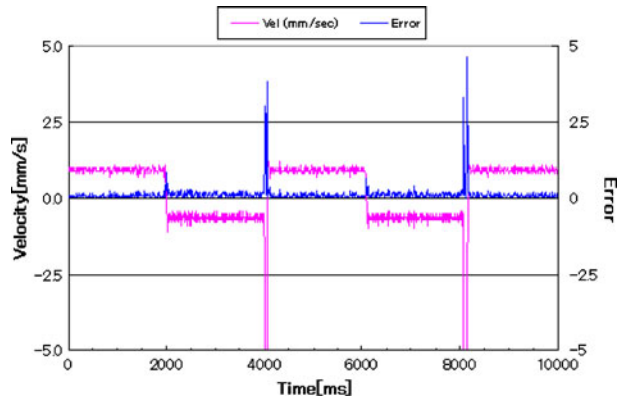
This detection system is connected to a stage controller of X–Y stage (SIGMA TECH), which realizes the positioning of the X–Y stage within 10 nm. The LIST-mode system saves (1) three different inputs from the detectors every 5 ns as function of time, (2) a start pulse from the function generator for the Mössbauer driver, and (3) a trigger signal from the stage controller which will be sent to A3100 when the positioning of the stage is achieved after moving the position from one to the next mapping position. Furthermore, the LIST-mode module (A-3100) accepts different timing signals for coincidence measurements, i.e., one fast-timing signal from a plastic scintillator placed around the ^{57}Co source to measure 122 keV- γ -rays and another TTL signal from a function generator which produces a blanker pulse for the SEM electron beam.

The SEM can be operated from the operation sub-panel, which is presently developed by APCO. The acceleration voltage of SEM can be varied between -0.5 and -5.0 kV, and the resolution is expected to be 15 nm at -5.0 kV with a beam current of 0.2 nA. This electron gun can be also used to inject electrons into a sample, and a mapping image can be simultaneously measured to study an electron induced phenomena. In addition, we detect the characteristic X-rays and the electrons due to Mössbauer effect alternatively, producing different mapping images.

(3) A new Mössbauer driver.

We are trying to use “a moving coil actuator with a liner encoder (SMAC)” as a new Mössbauer driver, the price of which is much cheaper than those of conventional Mössbauer drivers. Figure 3 shows the velocity of the source (red) as well as the error signal (blue) as functions of time obtained in a test run. By counting the electrons and γ -rays at the different velocities alternatively it is possible to measure the mapping images simultaneously corresponding to the different Mössbauer spectral

Fig. 3 The constant velocities of source are obtained alternatively at -0.63 and 0.93 mm/s, making us possible to measure the mapping images simultaneously corresponding to different spectral components



components. This actuator provides a positioning resolution of $1.0\mu\text{m}$, enabling us to move the ^{57}Co source either with a constant velocity mode, or with a constant acceleration mode. The former mode enables us to control the velocity within about 3 %, as can be seen in Fig. 3, while the latter (not shown in Fig. 3) within 10% depending on the magnitude of the maximum velocity presently. To improve the resolution, we are planning to use a different actuator with a higher resolution of $0.1\mu\text{m}$ as the next step.

2 Summary

The system and technology of “Mössbauer Spectroscopic Microscope” has been shown in this paper. Mainly described are the operation system based on LabVIEW program, the measuring system with a LIST-mode module combined with the X-Y stage controller and the new Mössbauer driver using a moving coil actuator with a liner encoder. The Mössbauer Spectroscopic Microscope appears to be promising to open totally new applications in the spectroscopy even in a laboratory.

Acknowledgements This project has been supported since 2008 by Japan Science and Technology Agency in the project: The development of systems and technology for advanced measurement and analysis.

References

1. Yoshida, Y., Suzuki, K., Hayakawa, K., Yukihira, K., Soejima, H.: Mössbauer spectroscopic microscope. *Hyperfine Interact.* **188**, 121 (2009)
2. Yoshida, Y., Kamimura, T., Ichino, M., Hayakawa, K., Yukihira, K., Soejima, H.: Mössbauer spectroscopic microscope. *J. Phys.: Conf. Ser.* **217**, 012003 (2010)
3. Yoshida, Y., Hayakawa, K., Yukihira, K., Ichino, M., Akiyama, Y., Kumabe, H., Soejima, H.: *Hyperfine Interact.* **198**, 23 (2010)
4. Soejima, H.: Japan Patent 2014379 (1986); Japan Patent 2001797 (1988)