

History of JEOL Microbeam Analysis: High Accuracy Analyses for Scientific and Industrial Work from the Centimeter to Nanometer Scale

Hideyuki Takahashi¹, Hiroyuki Yamada¹, Satoshi Notoya¹, Masaru Takakura¹, Takanori Murano¹, Vernon Robertson² and Peter McSwiggen²

¹ JEOL Ltd., 1-2 Musashino, 3-chome, Akishima, Tokyo, Japan.

² JEOL USA, 11 Dearborn Rd. Peabody, MA, USA.

JEOL Ltd. was established in 1947. Its first president, Kenji Kazato, and others developed its first product, a transmission electron microscope (TEM) [1]. About 10 years later, the first leader of the microbeam analysis division, Dr. Shizuo Kimoto, and other had started to develop a proto-type electron probe microanalyzer (EPMA), the JXA-1, by attaching wavelength dispersive spectrometers and an optical microscope to a transmission microscope, the JEM-5Y (Fig. 1). In 1960, members of Japanese electron microbeam society (The 141th Committee on Microbeam Analysis of Japan Society for the Promotion of Science) invited professor Raimond Castaing to Japan as a visiting scholar. This proved to be a great moment in the development of electron microbeam analyses within academia and industry in Japan. During the past 60 years in JEOL, many new spectrometers and electron optical systems, as well as application software, have been designed and developed through the cooperation of many scientists and industrial engineers.

The first JXA-2 had large WD spectrometers with a low take-off angle and a very high vacuum system, using dual diffusion pumps. The first real commercial EPMA for JEOL was the JXA-5 that was developed in 1967. During the same period, the first scanning electron microscope had also been developed. The last versions of the JXA-5 included a computer system for more accurate and faster quantification. Development of the EPMA was accelerated by the push from geological scientists who were striving for the best, most complete, and accurate analyses of lunar rocks collected through the historic Apollo project. In order to image and analyze extremely small phases, a much more sophisticated design of the electron optics was required. This resulted in the development of the so-called “mini-lens” that became part of the JXA-733 shown in Fig. 2 [2]. That instrument also had a very sensitive backscattered electron detector that could distinguish differences in the average atomic number of phases that were very similar to each other. Because of the critical requirements within industry to detect and measure the distribution for trace elements like carbon, manganese, silicon, phosphorous and sulfur in steel over large areas, mapping system using stage movement was developed and incorporated into the JXA-733. Once this was developed, applications followed for particle analyses, allowing for many thousands of inclusion and/or segregation in metals to be identified and analyzed. Because of these increased computational requirements, more powerful computer systems using mini-computers had to be adopted. To improve the sensitivity to detect trace amounts of very light elements, the layered dispersion element (LDE), synthetic analyzing crystals, was introduced. Changes in spectrometer design included one with a smaller Rowland circle, with only a 100 mm radius. It was developed to greatly increase the X-ray intensities. More recently, a large crystal spectrometer has also been developed to have both better energy resolutions, along with a high-count rate. In order to decrease analytical time, combined WDS/EDS systems were developed for the JXA-8621 series, and JEOL started to develop its own EDS systems. Recently, very large JEOL SDDs have been attached to TEMs to observe elemental distributions at the atomic scale. Beginning with the JXA-8800/8900 series, workstations with a user friendly GUI were introduced. Most recently, JEOL developed a parallel WDS detection system for soft

X-ray emission detection in collaboration with professor Terauchi, Dr. Koike and Shimadzu. Using varied-line space grating technology, without using a Rowland circle system, soft X-ray spectra with very high-energy resolution can be obtained (Fig. 3). It is possible to detect lithium, and can be used for chemical state analyzes. Mr. MacRae *et al.* at CSIRO is now working on the ultimate system combining EDS, WDS, CL and SXES into one system to characterize materials using all of these input sources [4]. In the future, automated system using these various spectrometers will be both very easy to use and will provide highly accurate material characterization for routine work.

References:

- [1] “Souzou to Kaihatsu” (Japanese), Creation and Development in JEOL Ltd. 60 Year Anniversary” (2010).
 [2] J. B. Reed, “Electron Microprobe Analysis second edition”, (Cambridge University Press).
 [3] M. Terauchi *et al.*, *J. Electron Microscopy* **61** (2012), p. 1.
 [4] C.M. MacRae *et al.*, *Microscopy & Microanalysis* **22(S3)** (2016), p. 410.

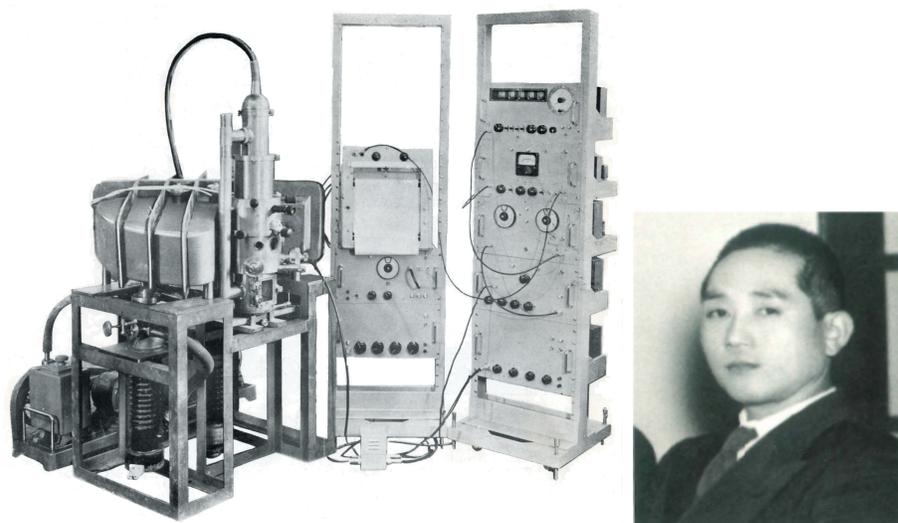


Figure 1. First JEOL EPMA, the JXA-1 (1960) (left) and developers; First JEOL president, Mr. Kenji Kazato (in his 28 years old).

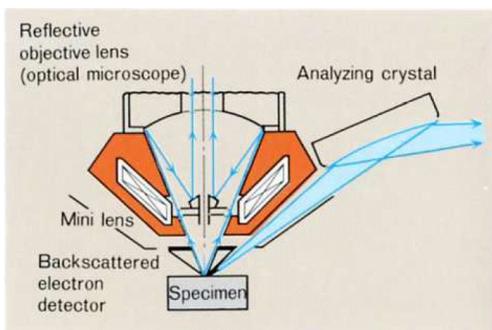


Figure 2. Illustration of the mini lens first developed for the JXA-733.



Figure 3. The JEOL EPMA JXA-8320 with attached WDS, EDS and SXES.