*Transactions of the Korean Nuclear Society Spring Meeting*
*Jeju, Korea, May 29-30, 2014*

# Development of a Quantitative Analysis Program for WDS-SEM

Hyoung-Mun Kwon*, Hang-Seok Seo, Wan-Ho Oh, Dosik Kim
*Korea Atomic Energy Research Institute, 1045 Daedeok-daero, Yuseong-gu, Daejeon 305-353, South Korea*

## 1. Introduction

WDS(Wavelength Dispersive Spectrometer) with an electron beam has been applied in various research areas, and is an essential instrument for analyzing the quantity of fission products inside a PWR fuel pellet. WDS installed in an ordinary SEM(Secondary Electron Microscope) is not optimized for a quantitative analysis, differently from EPMA(Electron Probe Micro-Analyzer).

Electron beam stability is very important for a quantitative analysis of a WDS-SEM [1]. We have developed a quantitative analysis program for a WDS-SEM including the function of inspecting the beam stability.

## 2. Methods and Results

In this section, some of the functions of the quantitative analysis program and the analysis procedure are described.

### *2.1 Quantitative analysis procedure*

Before and after measuring the X-ray intensity at a sample, the electron beam current is measured at the Faraday cup. The X-ray intensity at the center of a target X-ray peak is measured for a specified time, and then the X-ray intensities at the left background and the right background are measured for each half of the time. Figure 1 shows the procedure of the quantitative analysis by WDS-SEM.

When a relative standard deviation of a beam current is below 1%, the peak count rate is considered to be effective. The peak count rate is determined by the following equation:

$$C = \frac{C_p - \left(\frac{C_{lb} + C_{rb}}{2}\right)}{I}$$

where C is the peak count rate normalized by a beam current in cps/nA, $C_p$ is the peak count rate in cps, $C_{lb}$ is the count rate in the left background in cps, $C_{rb}$ is the count rate in the right background in cps, and I is the beam current on a Faraday cup in nA.

The expanded uncertainty of the peak count rate is calculated by the flowing equation:

$$U = 2\times\sqrt{\left(\frac{s_p}{\sqrt{t_p}\times I}\right)^2 + \left(\frac{s_{lb}}{\sqrt{t_b}\times 2I}\right)^2 + \left(\frac{s_{rb}}{\sqrt{t_b}\times 2I}\right)^2 + \left(\frac{C_p - \left(\frac{C_{lb}+C_{rb}}{2}\right)}{\sqrt{t_I}\times I}\right)^2}$$

where $s_p$ is the standard deviation of $C_p$, $s_{lb}$ is the standard deviation of $C_{lb}$, $s_{rb}$ is the standard deviation of $C_{rb}$, $t_p$ is the dwell time at peak, $t_b$ is the dwell time in the background, and $t_I$ is the dwell time of the beam current measurement on a Faraday cup

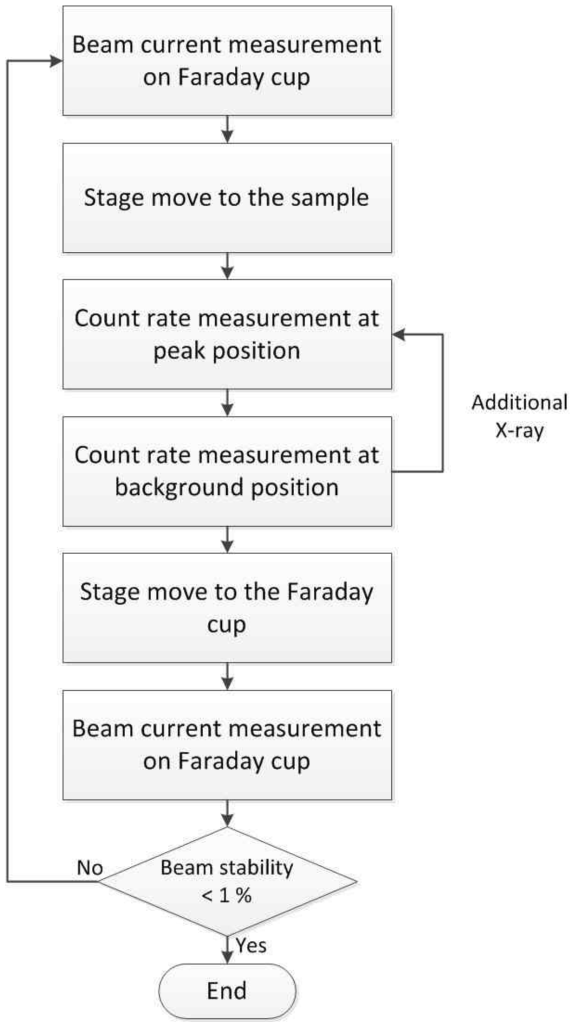


Fig. 1. Flow chart of WDS analysis procedure.

### *2.2 Functions of the program*

The program consists of a main view, a beam current monitor, and a COM port monitor. The main view has the functions of inspecting the WDS and beam current data, showing a graphical chart for the synchronized data of the count rate and current data, evaluating the peak count rate normalized by a beam current and determining the effectiveness of the data.

The beam current monitor has the functions of setting up the picoammeter, which measures the beam current, and confirming the values of the current. The COM port monitor shows information from the WDS such as the command and measurement data. The text in the window of the COM port monitor can be dragged and copied to a text editor.

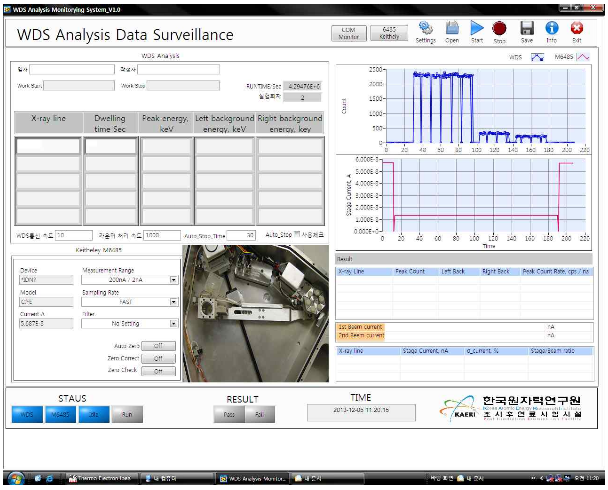


Fig. 2. Main view of the program.

### 3. Conclusions

This program will be applied to analyze the Xe behavior inside the irradiated nuclear fuel pellet and can be helpful to the uncertainty determination of the quantitative analysis by WDS-SEM.

### REFERENCES

[1] Anwar Ul-Hamid et al., Quantitative WDS analysis using electron probe microanalyzer, Materials Characterization, Vol.56, p.197, 2006