Elemental microanalysis using Silicon Drift Detector- Energy Dispersive X-ray Spectrometry (SDD-EDS): Spatial high spectral resolution in Scanning Electron Microscope (SEM)

Anit Dawar[1], *Leema Saikia[1], Meenakshi[1], Deeksha Khandelwal[1], and *G R Umapathy[1]

[1]*Inter University Accelerator Centre (IUAC), Aruna Asaf Ali Marg, New Delhi - 110067, India*

** email: umagrphysics@gmail.com, leema.saikia@msc2015.iitgn.ac.in*

Introduction

Electron excitation based X-ray spectrometry (i.e. EDS) is an established technique for the microanalysis of materials structure. SDD and Lithium drifted Silicon Si(Li) detector are X-ray radiation detectors used in X-ray spectrometry. SDD-EDS is rapidly erasing outperformed traditional Si(Li) detectors in almost every aspect [1]. Main advantages of SDD-EDS over Si(Li) are, higher count rates, higher energy resolution, Peltier cooling, and low level detection [1].In this paper, we are describing how resolution depends upon accelerating voltage and how we can determine the resolution of SDD.

SDD-EDS

X-ray is generated when an orbiting electron is displaced by electron microscope beam. X-ray emitted from different atoms are converted into electrical signals by SDD detector.X-ray is first detected and converted into charge and charge is then, converted into voltage signal. The pulse processor then measures the electronic signals into energy of each X-ray detected. Finally, analyzer display and interpret the data. Components of an EDS-SDD detector (Fig. 1) includes following arrangements [1].

1. Collimator assembly: It helps to maintain the limiting aperture so that only X-ray from the excited parts by electrons reach the detector and other X-rays are omitted [1].
2. Electron trap: The electrons can be deflected by a permanent magnet named as electron trap. It omits any stray electrons that can be made by passing through the detector [1]
3. Window: It is simply an obstacle window that is transparent to low energy X-rays (~100eV) and maintains a vacuum inside the detector [1]. It is made up of Mylar available in different thickness, referred as polypropylene.

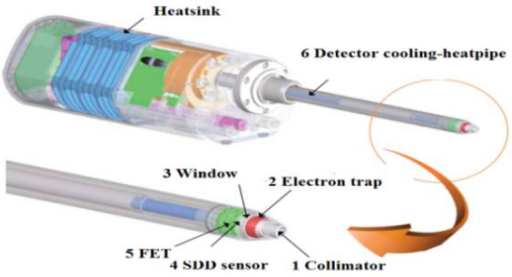

Fig.1: Components of SDD [1]

4. Sensor: It is a semiconductor (fig. 2) which usually converts each incident X–ray of certain energy into electrical charge through process of ionization [1].
5. FET: It provides pre amplification process where it measures the charge and converts it to voltage.
6. Detector cooling: SDD detectors are cooled by Peltier devices [1]. These devices are bounded to the SDD sensor which reduces the electrical noise of the sensor.

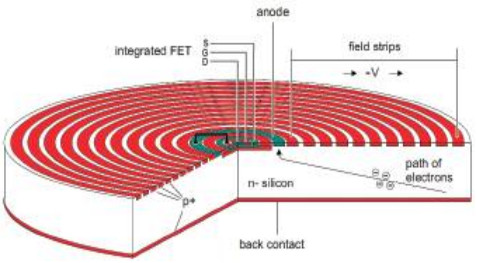

Fig. 2: Cross-section of circular SDD Sensor [2]

Why resolution is so important?

Better resolutions are important in the case where the two X-rays are very close that can happen in the case where target has more than one compound. Suppose, M X-ray energy of higher Z element can be close to M X-ray energy

of lower Z element. In this case having better resolution can benefit in elemental analysis. If the difference of the peaks between two energies is equal to or less then the instrument full width half maxima (FWHM) then, the two peaks got combined and show only one broader peak in place of the two individual peaks. The width of the FWHM thus increases with the failure in separating the two peaks and resolution got decreased. Thus, Lower the FWHM better the resolution.

The resolution $R(E_0)$ can be given by the formula, $R(E_0) = FWHM/E_0$ [3], where E_0 is energy.

Methodology

Traditionally, energy resolution for Si based detectors (SDD) is measured by using Manganese (Mn) Kα line [1].

Chemical analysis of pure Mn under the electron beam was performed with JEOL JSM 7610F using EDAX's Octane Plus (Ametek). Fig. 3 displays the EDS spectra of pure Manganese (Mn) on carbon tape. EDS spectra is collected with 9nA probe current, live time of 30s and at different accelerating voltages (10 kV, 15 kV, 20 kV, 25 kV and 30 kV). FWHM have been collected from the peak width of Mn-Kα line.

Results and Discussion

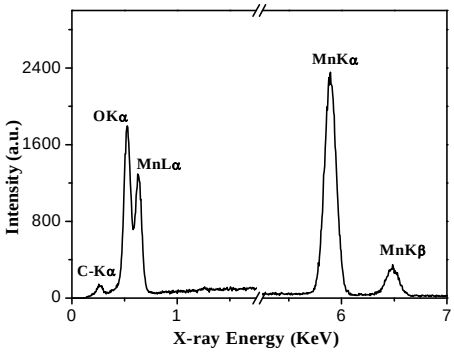

Fig. 3: EDS spectra of pure Mn at 15kV

From EDS (fig. 3), presence of Mn-Kα along with other element Carbon (C-Kα, due to carbon tape) and Oxygen (O-Kα, due to surface oxidation) is observed.

Table 1 shows dependence of FWHM and resolution with the accelerating voltages. Table 1 and Fig. 4 clearly depicts that with increasing accelerating voltages, line intensity increasing as well as FWHM which further increases the electron penetration and make the spatial spectral resolution worse. The weight (%) of Mn which varies with accelerating voltage is due to change in penetration depth.

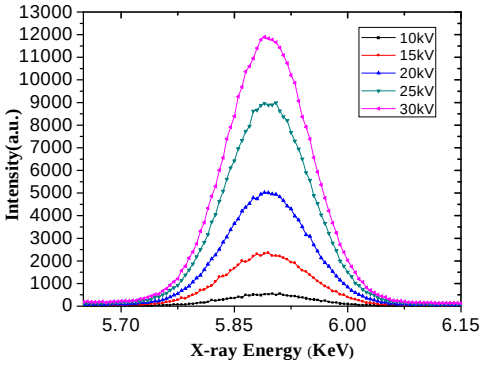

Fig.4: FWHM of Mn-Kα at various voltages

Voltage (kV)	Weight (%)	X-ray energy(keV)	FWHM (eV)	Resolution (%)
10	85.43	5.90	107.30	1.82
15	91.18	5.895	110.24	1.87
20	92.16	5.895	110.40	1.87
25	95.73	5.890	111.13	1.89
30	98.64	5.890	111.05	1.88

Table 1: weight (%) and FWHM of Manganese at different voltages.

Conclusion

Physical aspect of SDD for operation and functioning is discussed. It must be noted that the accelerating voltage must be twice the highest excitation energy of an element. The accelerating voltage should be at least between 15-20kV for prominent spatial spectral resolution.

Acknowledgment

Authors are thankful to Dr. D. Kanjilal for valuable scientific discussion.

References

[1] https://nano.oxinst.com/assets/uploads/products/nanoanalysis/documents/Brochures/SDD%20Explained.pdf

[2] https://assets.thermofisher.com/TFS-Assets/CAD/Warranties/TN52342_E_0512M_SiliconDrift_H.pdf

[3] Measurement and Detection of Radiation. Editors: Nicholas Tsoulfanidis and Sheldon Landsberger, 4th edition, CRC Press, Taylor & Francis Group, Boca Raton, 2015.