LABORATORY 2

ELEMENTAL ANALYSIS BY X-RAY SPECTROSCOPY

A major application are of X-rays is spectral measurements, i.e. chemical analysis of materials in elemental form. The basis for this analysis is that when samples are bombarded with X-rays or electron beams of high enough energy, elements that make up the sample generate their characteristic radiations.

2.1. Introduction

In the previous experiment, you have seen that any element (the target of an X-ray tube) emit their characteristic lines (Kα, Kβ, etc.) when bombarded with electrons of high energy. They are called as "characteristic lines" to emphasize the fact that their wavelengths are fixed and characteristic of the emitting element.

You have also seen that the same lines would be emitted if the element were bombarded with the X-rays with high enough energy. In that case, the radiation emitted is called as "fluorescence radiation", which was mentioned previously within the context of "X-Ray Absorption" in lecture hours.

Therefore, in order to carry out elemental chemical analysis, two approaches are possible. The first approach is to use an X-ray source (white radiation, preferably W-tube with a power rating as high as possible), see Fig.2.1, and the second approach is to use an electron beam in an Electron Column Instrument (Microprobe, Scanning Electron Microscope, or Transmission Electron Microscope).

Then, the wavelengths (characteristic lines) generated by the sample either exposed to an X-ray beam or an electron beam can be analyzed by "Wavelength Dispersive Analysis" or "Energy Dispersive Analysis".

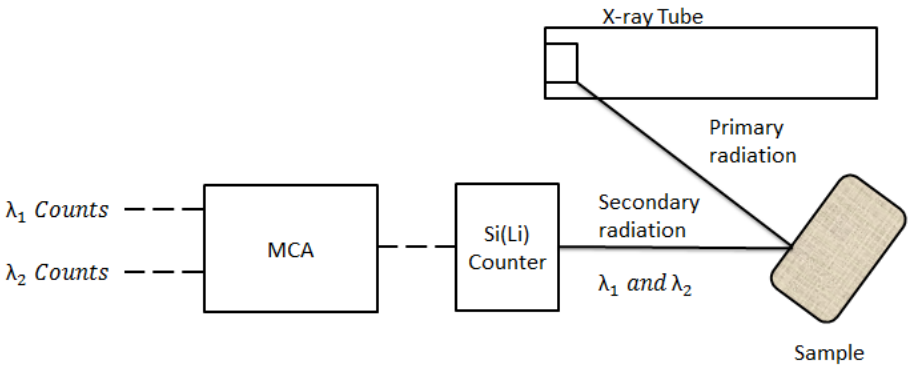

Fig.2.1 Schematic representation of spectroscopy with energy dispersive system.

2.2. Energy Dispersive Analysis

Energy dispersive system is quite common in electron column instruments, such as scanning electron microscope (SEM) is given in Fig. 2.2. The sample is bombarded with electrons of high energy and the radiation emitted by the sample is then analyzed by the use of an energy dispersive system. This can be achieved with the use of counters, which generate pulses with heights proportional to the energy of the X-ray photons received. Semiconductor counters such as Si (Li) counter is quite suitable for this purpose.

The counter is used in conjunction with a multi-channel analyzer (MCA). MCA is made up of a large number of channels, which cover the complete energy spectrum. Each channel responds to a narrow window within that spectrum. Since all energies are accumulated simultaneously, the time interval over which the X-rays are counted will not be long.

Experiments

In this experiment, powder samples, namely XA, will be examined in a Scanning Electron Microscope (SEM). A schematic representation of SEM is given in Fig.2.2. The instrument consists of an evacuated column with facilities that enable electron beam to be focused to a small spot (approximately 100 Å).

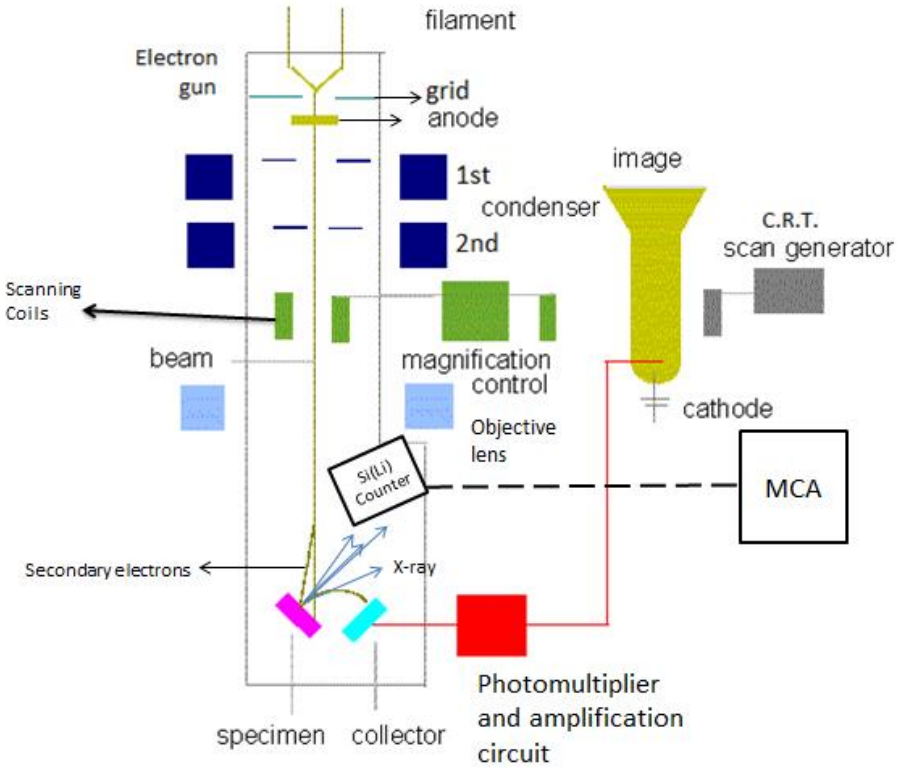

Fig.2.2 Schematic of a scanning electron microscope (SEM)

In general, SEM is used as a microscope. The image is formed by scanning the electron beam is over the sample surface. The counter, the output of which is fed to a video display unit (VDU), then collects the electrons ejected from the sample surface (secondary electrons). Scanning coils of the VDU and scanning for electron beam is synchronized, so that the “image” of the sample surface appears on the screen.

For chemical analysis, an energy dispersive system can be activated. This system consists of Si (Li) counter and MCA. The content of MCA is displayed in the VDU in the form of chart intensity vs. energy (in units of keV) see Fig.2.3, from which the elements present in the sample can be identified. The elements present can also be quantified.

Several modes of analysis are possible. Spot analysis is achieved by directing the electron beam to a particular point on the sample. On the other hand, average analysis can be obtained by operating the microscope in normal, i.e. scanning mode. Specific channels in MCA can be made the basis of “elemental mapping” which is useful in the context of multiphase materials and for study of segregation.

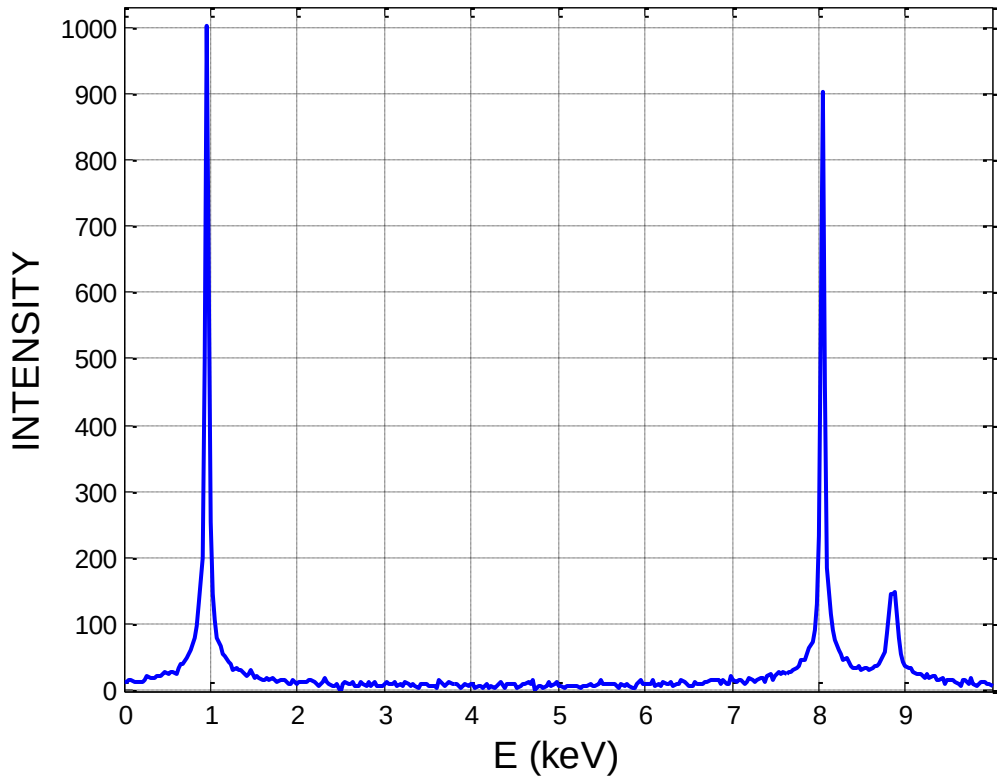

Fig.2.3 Elemental analysis of copper powder with energy dispersive system (EDS).

HINT: Elemental Analysis by EDX

By performing elemental analysis with energy dispersive system (EDS), you obtained intensity versus energy graphs for your unknown powder sample. On these graphs, there are several peaks, each indicating a characteristic radiation of an element existing in that sample. In order to find out element(s) present in your sample, follow the steps given below:

1. Label each peak from left to right as "Peak 1, Peak 2, ...".
2. Locate the center of the each peak through the x-axis (energy axis).
3. Through the horizontal axis (which denotes the energy), each spacing indicates 1 keV. Therefore, measure the distance between the origin and the center of the peak to find out the energy of the peak in question. Repeat this procedure for each evident peak.
4. Convert eV (or keV) to Joules (or kJ).
5. Calculate the corresponding wavelengths (λ) of these energy values (in terms of joules) by using the equations "$E = h\nu$" and "$\nu = c/\lambda$".
6. Compare the calculated wavelength values with the values given in the Appendix 7: "X-Ray Wavelengths" in order to determine the element(s) present each sample.

Self-Study: Elemental Analysis by EDX

1. Evaluate the below spectrogram by using the procedure given in laboratory section and determine the elements present in this sample.

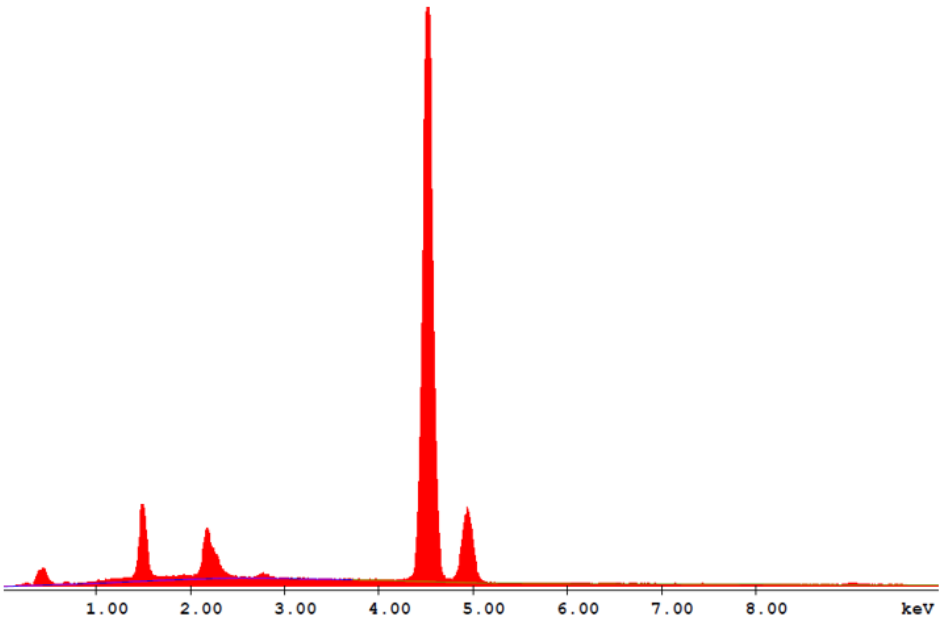

Fig.2.4 Energy dispersive system (EDS) of unknown samples.