Development of Wavelength Dispersive X-ray Spectrometer

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A linear type wavelength dispersive X-ray spectrometer is designed, fabricated assembled, and tested for elemental analysis. Four analyzing crystals (mounted on a turret) cover the detection range from at. no. 5 (boron) to at. no. 92 (uranium). A digital electronic control system is designed and tested for setting the crystal position with the help of a stepper motor. The motion of the turret on lead screw can be controlled to a high degree of accuracy with the electronic control system. It is possible to scan the X-ray spectrum range, as mentioned above. The ability to scan gives easy operational flexibility and opportunity to search over an extensive wavelength range. Samples of brass, stainless steel, and mixture of five adjacent elements (Fe, Co, Ni, Cu and Zn) are analysed by this instrument. X-ray analysis peaks of different elements present in the sample are recorded on a strip chart recorder and well resolved. This instrument can be utilized for X-ray fluorescence or as an integral part of scanning electron microscope (SEM) for elemental analysis.

Introduction

X-ray spectrometer is an integral part of a modern scanning electron microscope (SEM) for microanalysis purposes. As the high-energy electron beam impinges on a specimen, electron-specimen interaction takes place. Some of the typical interactions are secondary electrons, characteristics X-rays, back-scattered electrons, specimen current, etc. Their interactions can be utilized for specimen imaging as well as elemental analysis. Secondary electrons provide the best possible signal for surface topographic studies and the emission of characteristics X-rays is frequently used for material characterization. By measuring the wavelength of the X-rays and comparing their intensities with suitable standards, elemental composition in microvolumes of the specimen can be determined. A SEM, which gives details of surface topography with great depth of focus and high resolution when fitted with X-ray spectrometric system also reveals micro-composition and highly accurate material characterization is also possible.

The wavelength and the intensity of the characteristics X-ray photons emitted from the specimen are measured by X-ray spectrometer. The X-ray spectrometer can be either wavelength dispersive (WD) or energy dispersive (ED). The ED X-ray spectrometry differs from the WD spectrometry only in the methods used to disperse the several spectral lines emitted by the specimen. This gives rise to major difference in the types of instrumentation used in the above techniques. In the energy dispersive spectrometer all emitted lines of the specimen elements fall on the detector simultaneously. The detector consists of a solid-state semiconductor, lithium drifted silicon detectors Si (Li), and its preamplifier contained in a vacuum cryostat cooled by liquid nitrogen. It converts each absorbed X-ray photon into a current pulse whose amplitude is proportional to the energy of the characteristic X-ray photon. Qualitative and quantitative energy dispersive analysis is carried out on the basis of pulse heights and their intensities respectively. In the WD (crystal) spectrometers, several wavelengths are separated on the basis of their wavelengths before detection. Thus, an X-ray photon of one wavelength is detected by the proportional counter at a time. The resolution of the WD spectrometer is better than that of the ED spectrometer.

Materials and Methods

Mechanical System for Linear X-ray Spectrometer

The basic principle of the linear X-ray spectrometer is shown in Figure 1. The wavelength
dispersive spectrometer complies Johann type focusing arrangement. The mechanism developed moves crystal and detector on the Rowland circle so that Bragg's condition \( n\lambda = 2d \sin \theta \) is fulfilled at all points for different \( \lambda \), where \( \lambda \) is the wavelength of x-ray, \( d \) is the interplaner space, \( \theta \) is the Bragg angle and \( n \) is the order. The focusing requirement is maintained by moving the analyzing crystal along a straight path, on a lead screw, away from the source and rotating the crystal simultaneously. The angle \( \theta \) and the linear displacement \( L \) are related by the equation \( L = 2R \sin \theta \), where \( R \) is the radius of the focusing circle. It comes out that crystal to source distance is directly proportional to the wavelength \( A = dRL \) and this distance is calibrated in terms of wavelength. The crystal is automatically positioned accurately by a digital electronic control system with the help of a stepper motor. Figure 2 shows the working of the various mechanical parts of the spectrometer. The Rowland circle covers Bragg angle from 15 to 75°. The spectrometer is provided with a turret to hold four analyzing crystals of a different 'd' values, interchangeable in vacuum with provision for fine adjustment of Bragg angle setting accurately for each crystal independently. The ingenuity of the system lies in coupling the linear slide motion on the lead screw and the curve linear assembly with the help of belt and pulley arrangement to achieve the desired performance. The Rowland circle diam is 300 mm and the analyzing crystals are bent at this radius with indicated wavelength range and dispersion are mentioned in Table 1.

![Figure 1 — Semi-focusing crystal spectrometer geometry](image)

<table>
<thead>
<tr>
<th>Material</th>
<th>( A ) (( \text{mm} ))</th>
<th>( \text{mm/} )A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Topaz</td>
<td>0.3 – 2.5</td>
<td>125</td>
</tr>
<tr>
<td>LiF</td>
<td>0.4 – 3.8</td>
<td>75</td>
</tr>
<tr>
<td>KAP</td>
<td>3 – 25</td>
<td>10</td>
</tr>
<tr>
<td>Pb St</td>
<td>9 – 95</td>
<td>3</td>
</tr>
</tbody>
</table>

The Rowland circle locus moves on a circular track. To maintain 1:2 ratio in the angular motion of the crystal and detector arms, pulleys of 2:1 ratio having 300 mm diam have been used and rotation is given with a steel tape, tensioned by a spring. The alignment of crystal to counter is carried out by a sliding link arrangement. From the crystal x-rays of selected wavelength enter into the detector, which is a proportional counter. The signal from the detector is processed by the conventional electronic proportional counting system.

**Digital Electronic Control System**

A digital electronic control system has been designed for automatic operation of the spectrometer with the help of a stepper motor. The block diagram of the system is shown in Figure 3. The present figure (in terms of distance) computed from standard tables is stored in the storage registers through a decimal to BCD encoder circuit and then displayed by seven segment LED displays. Also, digital pulses of varying frequencies (500 to 10,000 pulses/min) are generated from a crystal oscillator and a dividing network to vary the motor speed. These pulses of selected frequency are counted and then compared with the present value by four bit magnitude comparators. When the two values equalize the resulting output signal closes the control gate, which in turn blocks further passage of pulses to the stepper motor. This stops the stepper motor and hence the spectrometer instantly. Apart from displaying the final set point, provision has been made to have a second display to ascertain the actual position of the crystal during motion. The present system is capable of presenting three digits before as well as after the decimal point thus giving a high degree of accuracy. Lowest speed of 2.5 rev/mm is used for actual recording the x-ray spectrum on a chart recorder, whereas higher speeds are useful while searching the unknown elements by quickly scanning the whole range.
The turret assembly of the x-ray spectrometer is driven to either side on a lead screw by a stepper motor. In order to prevent damage to the stepper motor/spectrometer limiting switches are provided. The turret assembly on reaching each of the extremities operates a pair of micro switches. The micro switches stop the stepper motor when the extreme limit is reached in clockwise direction, the limit switch operates and stops the clockwise rotation of the motor. Supply voltage for the anti-clockwise
The output signal from a proportional counter is a quantity of charge. A charge-sensitive preamplifier has been designed and tested. The first stage is an FET input high gain Operational Amp operated in the charge-sensitive mode, since it integrates the charge on the feedback capacitor. The charge sensitive mode is preferred over the voltage-sensitive technique because its gain is not dependent on the detector capacitance or input capacitance. The charge sensitivity in the present case works out to be $0.20 \times 10^{12}$ V/C. The second stage is a buffer amplifier connected in a bootstrapping mode to increase the input impedance of the voltage follower. The low output impedance of the output stage of the preamplifier is suitable to drive a long coaxial cable needed for the main amplifier. The preamplifier is housed very close to detector mounted inside the spectrometer for better signal to noise ratio. The linear amplifier, single channel analyzer, ratemeter and strip chart recorder have been procured from ECIL, Hyderabad for use in the X-ray spectrometer.

The x-ray spectrum as, recorded on a strip chart recorder is shown in Figure 5 for mixture of adjacent elements Fe (26), Co (27), Ni (28), Copper (29) and Zn (30), Figure 6 for stainless steel and Figure 7 for brass. The x-ray peaks of the different elements present in sample are very well resolved. The
fluctuations/noise on the base line is, however, due to the unstabilized supply of the x-ray source.

**Main Physical Parameters of Wavelength Dispersive x-ray Spectrometer**

- **X-ray spectrometer**: A linear type semifocusing (Johann design) spectrometer with four crystals on a turret.
- **Take off angle of x-rays**: Variable up to 60°
- **Detectable elements**: Boron (5) to Uranium (92)
- **Diameter of Rowland circle**: 300 mm
- **Analyzing crystal**: LiF, Mica, Topaz, PbSt (Lead stearate)
- **Crystal size**: 10 mm x 25 mm
- **Crystal changing**: Crystals are mounted on a turret and can be interchanged under vacuum
- **X-ray detector**: Proportional counter
- **Collimation**: Primary defining aperture, secondary collimator located in front of x-ray detector window
- **Scanning speeds**: Switch selected, six standard speeds
- **Bragg angle (θ) range**: 15 to 75°
- **Mechanical drive system**: High precision stepper motor drive
- **Control for stepper motor**: Programmable with two digital displays

Figure 5 — X-ray spectrum of the mixture (Fe, Co, Ni, Cu, Zn) as recorded on strip chart recorder.

Figure 6 — X-ray spectrum of the stainless steel, as recorded on strip chart recorder.

Figure 7 — X-ray spectrum of the brass as recorded on strip chart recorder.
Results and Discussion

The performance of the instrument has been thoroughly checked and satisfactory working is established. X-ray spectrum of a mixture of five adjacent elements, (Fe, Co, Ni, Cu, and Zn) has been successfully recorded. Analysis of other different combinations of elements by fluorescence has also been carried out.

Conclusion

This instrument covers a wide range for elemental analysis, viz. starting from at. no. 5 (boron) to at. no. 92 (uranium). It has good resolution to resolve x-ray spectrum of adjacent elements like Fe, Co, Ni, Cu, and Zn, etc.

References