

## WEDAX STUDIES ON THE HITACHI S.E.M.

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### ABSTRACT

The WEDAX (Wavelength Dispersive Analysis of X-rays) system incorporated into the Hitachi S.E.M. is a method of performing chemical microanalysis of a material. The primary objective of this project was to bring up the system to full operating status and calibrate the WEDAX utilizing known samples of aluminum, copper, and silicon. As of May 1988, the S.E.M. resolution is at 2K and the WEDAX is still inoperable.

### INTRODUCTION

Wavelength Dispersive Analysis of X-rays (WEDAX) is a method of performing chemical microanalysis of a wafer surface. This technique is based on the theory that as the specimen is irradiated with an electron beam, x-rays are emitted with energies and wavelengths characteristic of the specimen's elemental composition. As the impinging electron beam strikes the surface atoms, electrons are inelastically scattered causing x-rays to be formed by two distinctly different processes. The incident electrons decelerate upon encountering the Coulombic field of the atomic core, which consists of the nucleus and tightly bound electrons, leading to the formation of a continuous spectrum of x-rays. This continuous spectrum of x-rays varies from an energy of zero up to the value of the incident electron energy [1]. The above process is referred to as 'bremsstrahlung', or "braking radiation." The continuum x-rays detected will show up as a form of background noise on the graph. The x-rays that we are primarily concerned with are those called characteristic x-rays produced by a sufficiently energetic beam electron causing an inner-shell electron, K, L, or M, to be ejected, leaving the atom in an ionized, or excited state. It takes the atom approximately ten picoseconds [2] to relax to its original ground state after ionization. In the process of relaxation, transitions of electrons from a higher shell to the vacancy, for example, from the L to K shell, results in the excess energy being released in the form of a photon of electromagnetic radiation. This x-ray emission process is summarized in Figure 1.

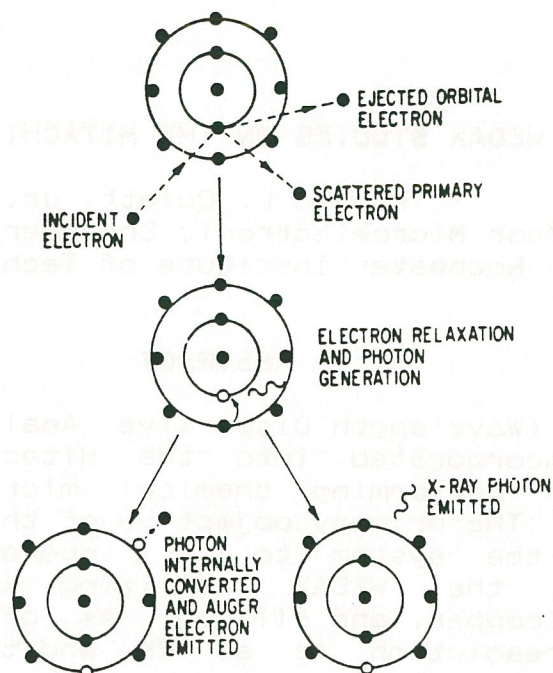


FIGURE 1: X-ray Generation Process. [2]

Various possible x-ray producing transitions are shown in Figure 2.

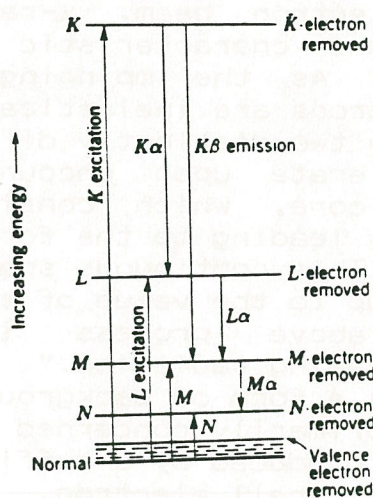


FIGURE 2: Electron Shell Transitions. [2]

A transition of one shell is denoted by the  $\alpha$  subscript and that of two shells by a  $\beta$  subscript. The energy of the photon is equal to the difference in energy between the shells involved in the transition, and for inner-shell transitions, the energy is such that the photon lies in the x-ray range of the electromagnetic spectrum.



These photons, according to the Duality Principle, have an associated wavelength which is related to the photon energy by:

$$\lambda = hc/qE$$

where  $h$  is Planck's constant,  $c$  is the speed of light,  $q$  is the electron charge in coulombs,  $E$  is the energy in keV, and  $\lambda$  is given in nanometers. Due to the discrete energy levels between shells, we can fingerprint an element by its characteristic x-rays which are well known and tabulated.

Detection of the x-rays is a straightforward matter employing Bragg's formula. A small portion of the x-rays generated from the sample pass through a port in the specimen chamber and enter the WEDAX housing. The x-rays impinge on the analyzing crystal, are diffracted and enter the detector, as depicted in Figure 3. If Bragg's law is satisfied:

$$n \lambda = 2 d \sin \theta$$

where  $n$  is an integer,  $\lambda$  is the x-ray wavelength,  $d$  is the lattice spacing of the analyzing crystal, and  $\theta$  is the incident angle of the x-ray on the crystal, constructive interference will occur, providing a maximum at the detector. The distance  $l_0$  from the sample to the analyzing crystal in the x-ray spectrometer can be expressed by:

$$l_0 = 2 r \sin \theta$$

where  $r$  is simply the Rowland circle radius of the spectrometer. Combining the two equations, we see that  $l_0$  is proportional to the wavelength by the equation:

$$\lambda = (d/r) l_0$$

Thus, with known crystal lattice spacings and a fixed Rowland circle radius, determining the wavelength is a simple matter of reading  $l_0$  from the dial of the spectrometer and performing a simple conversion.

FIGURE 3. Schematic Diagram of WEDAX and WEDAX System

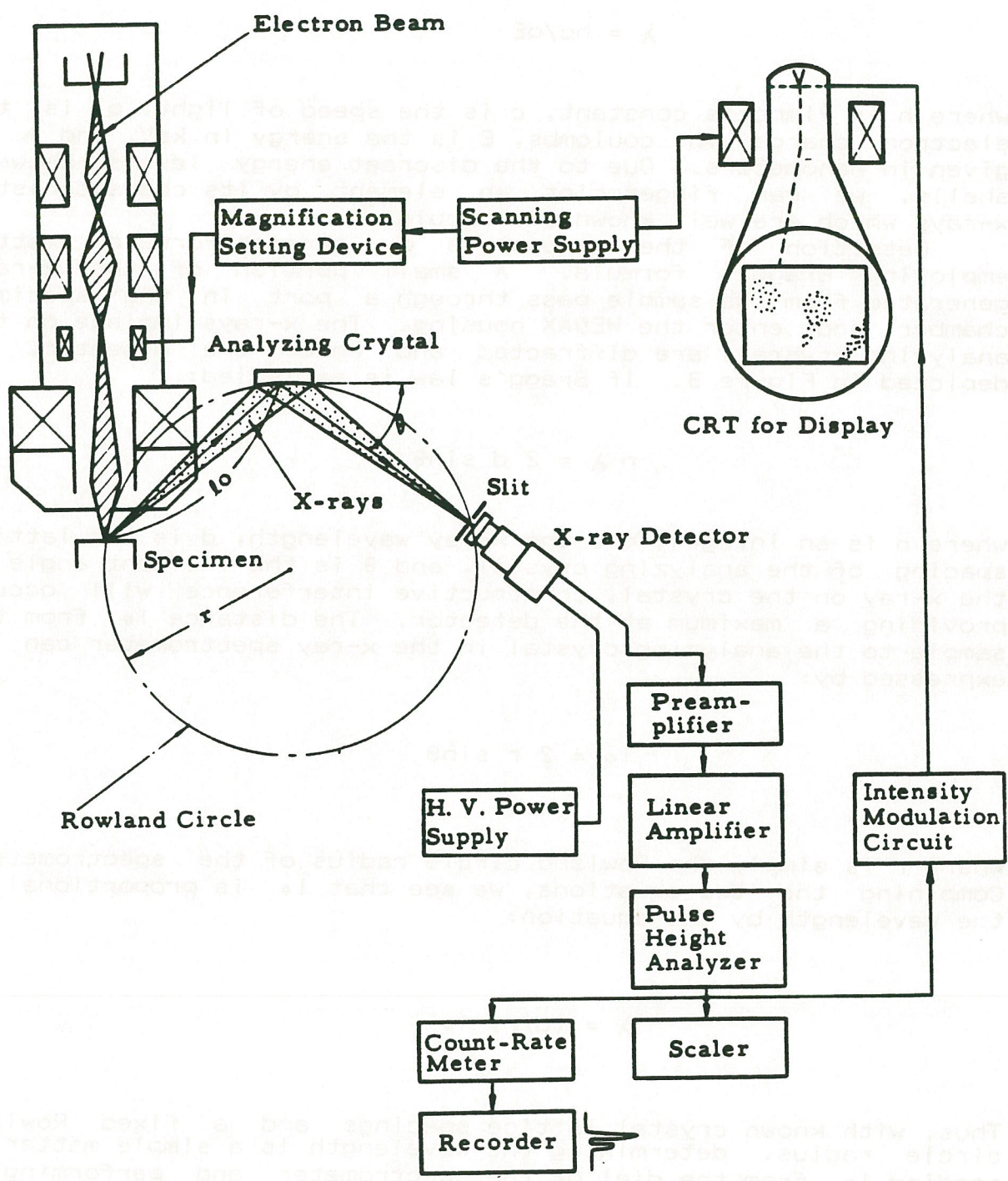


FIGURE 3. Schematic Diagram of Hitachi and WEDAX System. [3]



Upon entering the detector, the x-rays ionize the counting gas, a mixture of 90% argon and 10% methane, causing current to flow. This signal, proportional to the amount of x-rays detected is fed through a series of amplifiers, a pulse height analyzer, count rate meter and finally plotted on a chart recorder. The graph is a plot of the number of x-rays counted as a function of  $l_0$ , with the maximum peak corresponding to the major element contained in the sample. Figure 4 shows an example of a K $\alpha$  transition for a sample containing copper, where  $l_0$  is expected at 127.58 mm.

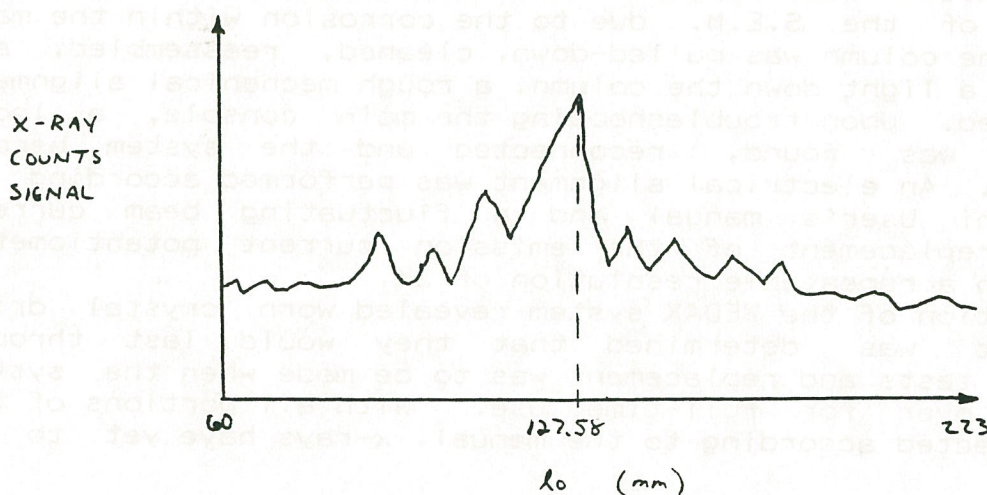


FIGURE 4. Chart Recorder Graph

## EXPERIMENT

The project was concerned with bringing up the S.E.M. and the WEDAX system. After connecting the utilities the system was ready to be powered up. Once running, the electrical and mechanical alignments were performed on the S.E.M. in accordance with the Hitachi User's Manual. Facilities for the WEDAX consisted of 110VAC and P-10 gas for the detector. P-10 gas is a mixture of 90% argon and 10% CH<sub>4</sub> and enters the system at a flow rate of 1 bubble per second as measured by an auxiliary tube from the gas manifold and placed in a beaker of isopropyl alcohol. A sample of copper was then affixed to a specimen holder and irradiated by the S.E.M. in the spot mode at an accelerating voltage of 30KeV. The x-ray detector signal was routed from the pulse height analyzer through the count rate meter on the EDAX system and into a 10K resistor network. At the resistor network the signal was converted from a voltage to a current enabling it to be fed into the chart recorder and displayed as a function of time.

With the chart recorder running, the crystal drive was started with an initial  $l_0$  reading of 60mm and ran to 223mm. The crystal used was RAP (rubidium acid phthalate) with a crystal



lattice spacing,  $d$ , of  $13.06\text{\AA}$ . The  $1\theta$  reading associated with the highest peak was then compared to the expected table value. If a discrepancy occurred, the crystal would be placed at the position of maximum signal occurrence and the  $1\theta$  indicator assembly adjusted to reflect the expected value. This entire process would be repeated for samples of aluminum and silicon, with the final positioning of the indicator being a 'best fit' setting for all three samples.

## RESULTS

Many hours were spent troubleshooting the electrical components of the S.E.M. due to the corrosion within the main console. The column was pulled-down, cleaned, reassembled, and by shining a light down the column, a rough mechanical alignment was performed. Upon troubleshooting the main console, a loose connection was found, reconnected and the system became operational. An electrical alignment was performed according to the Hitachi User's manual and a fluctuating beam current warranted replacement of the emission current potentiometer resulting in a repeatable resolution of  $2\text{K}$ .

Inspection of the WEDAX system revealed worn crystal drive gears. It was determined that they would last through preliminary tests and replacement was to be made when the system is turned over for full-time use. With all portions of the system connected according to the manual, x-rays have yet to be detected.

## SUMMARY

Further restoration of the Hitachi S.E.M. and WEDAX maybe questionable due to the excessive corrosion of all PC boards within the main console. If and when the WEDAX becomes operational, the replacement of the crystal drive gears is warranted. The S.E.M is functional.

## ACKNOWLEDGEMENTS

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## REFERENCES

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- [2] Goldstein, J.I., "Scanning Electron Microscopy and X-ray Microanalysis." Plenum Press, N.Y., 1981, pp. 95-122, 205-222.
- [3] Hitachi Users Manual, 1976.