

ENERGY DISPERSIVE SPECTROSCOPY X-RAY MICROANALYSIS

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ABSTRACT

An overview of Energy Dispersive Spectroscopy (EDS) X-ray microanalysis is presented with attention to artifacts present in the X-ray spectrum, such as the Bremsstrahlung, sum peaks, and silicon escape peaks. Sample spectra obtained from Al on Si, Cu on Si, and PZT ferroelectric films demonstrated the capability of obtaining qualitative elemental composition. The elemental spatial distribution, or the X-ray mapping, capability was also demonstrated utilizing a special alloy sample.

INTRODUCTION

During the past several years, a remarkable growth has taken place in the applications of analytical imaging instruments, such as Scanning Electron Microscopes (SEM) and Transmission Electron Microscopes (TEM), to support process development activities in the semiconductor industry. Among these techniques, X-ray microanalysis has emerged as one of the most valuable and essential tools.

X-ray microanalysis involves the identification of the X-rays emitted from a specimen excited by an electron beam. The analysis can be accomplished by either of two techniques; Energy Dispersive Spectroscopy (EDS) or Wavelength Dispersive Spectroscopy (WDS). In EDS, X-rays emitted from the sample are discriminated based on their energy level. For WDS, diffracting crystal spectrometers are used to select the particular X-ray wavelength of interest. Table 1 lists some of the advantages and disadvantages of each technique. In general, WDS is the method of choice when quantitative identification of elements is the principal goal of an investigation due to its ability to highly resolve wavelength. On the other hand, EDS is the method of choice when qualitative analysis is required in minimal time.

<u>Method</u>	<u>Advantages</u>	<u>Disadvantages</u>
EDS	Less expensive. High efficiency. Fast qualitative analysis. Analyze all elements simultaneously.	Resolution limit. Spectral artifacts. High background. Poor light element detection ($Z < 11$).
WDS	High spectral resolution. Low background. Good quantitative analysis. Good light element detection.	Slow qualitative analysis. Low efficiency. Expensive & complex. Analyze one element at a time.

Table 1: EDS vs WDS.

Figure 1 illustrates the phenomenon of X-ray generation in an atom upon electron beam excitation. If the beam energy is sufficient, inner shell electrons may absorb the energy and be photo-emitted. Because electrons tend to occupy the lowest available energy state, an outer shell electron may relax to fill the vacancy in the lower shell by releasing a photon of energy equal to the difference between its initial and final states. If the energy is large enough, the photon is in the X-ray regime.

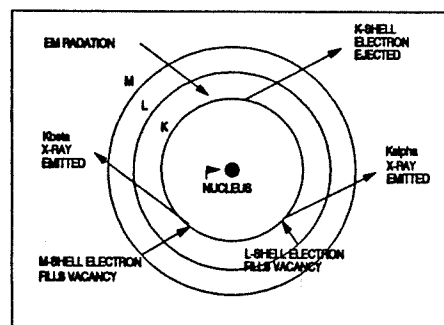


Figure 1: X-ray generation.

Two important points must be recognized from the above process. First, for a fixed electron acceleration potential, the L to K transition (K_{α}) is more probable than the M to K transition (K_{β}). Therefore, K_{α} radiation of a chosen element is always more intense than K_{β} radiation. Empirically, it has been found that K_{β} intensity is approximately 10% of the K_{α} , the L_{β} is approximately 70% of the L_{α} , and the M_{β} is approximately 60% of the M_{α} [1]. The second important point is that the

closer the final energy state is to the nucleus of the atom, the higher the energy of the emitted X-rays will be for an electron relaxing from a given initial state. Therefore, one should expect to see a series of K X-rays at higher energy locations than a series of L or M X-rays. Figure 2 illustrates these points.

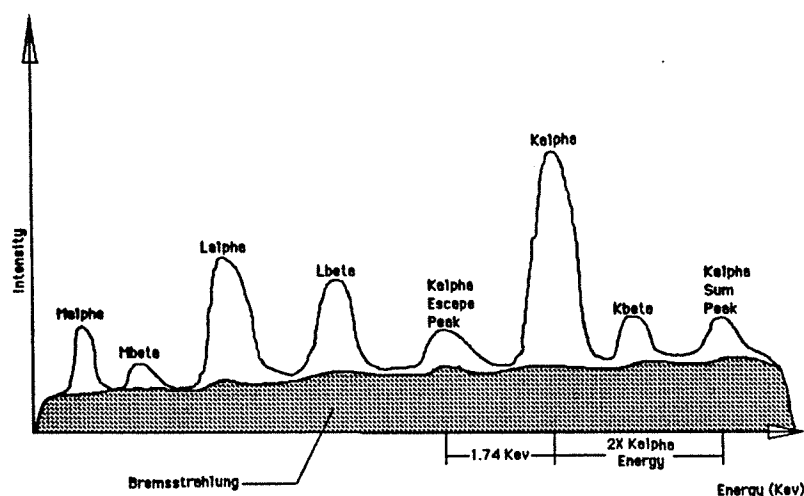


Figure 2: X-ray spectrum.

Figure 2 also illustrates some artifacts which must be considered. There is a second type of X-ray generation that occurs due to the electron deceleration within the material. These are non-characteristic X-rays because their energies are independent of the target material. This type of radiation spectrum is called Bremsstrahlung, and it constitutes background noise which can represent a significant portion of an X-ray spectrum. Because Bremsstrahlung occurs over a large portion of an X-ray spectrum, the identification of low intensity characteristic X-ray peaks becomes very difficult, hence limiting the sensitivity of an EDS system.

Other artifacts associated with EDS technique are pulse pile-up peaks and Si escape peaks. A pile-up peak occurs when multiple X-ray photons reach the detector at the same time, and the pulse processing electronics erroneously record the sum of their energies rather than individually. This results in a peak at twice the energy of the main elemental peak. A silicon escape peak occurs when an X-ray strikes the detector (a special Si diode) and generates Si Kalpha X-rays (with an energy of 1.74 Kev) in the detector. For the most part, these X-rays are reabsorbed by the detector so that the signal from the detector represents the energy of the specimen X-ray. However, there is a finite probability that Si Kalpha X-rays might escape the detector, thus reducing the specimen X-ray by 1.74 Kev. For example,

when Cu Kalpha X-rays with 8.04 Kev energy reach the detector, they will produce a characteristic peak at 8.04 Kev and an escape peak at 6.30 Kev (8.04-1.74 Kev). In principle, Si Kbeta X-ray photons with the energy of 1.832 Kev could also escape the detector. However, the probability for Kbeta formation is so low that it is rarely detected [2].

The critical issue concerning these artifact peaks is that pile-up peaks and Si escape peaks may become misassigned peaks, which should have been a part of the main elemental peak. Therefore, it is vital not to mistakenly identify pile-up and escape peaks as other elemental peaks.

Another common error is misinterpretation of composition due to the low lateral resolution of EDS. Looking at the ideal case depicted in Figure 3, it is desirable to have the beam-sample interaction volume narrow such that X-ray generation is confined to only material 2. However, in a real situation, electrons interact with the matrix of a material, producing a much larger volume of interaction. As a result, the collected X-ray spectrum will contain X-rays pertaining to both materials 1 and 2.

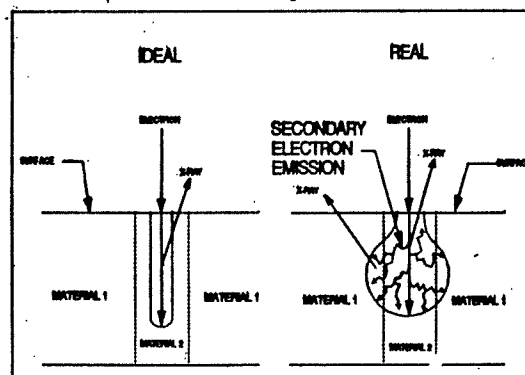


Figure 3: Secondary electron emission.

There are several parameters in EDS analysis that can be optimized in order to obtain more accurate results. Two SEM parameters are the electron acceleration potential and electron beam current. Generally, the accelerating potential of the electrons must exceed the critical excitation energy--a specific energy requirement necessary to excite a given X-ray line--of the target element by a factor of 1.5 to 3 in order to efficiently produce an X-ray. The accelerating potential that is too low reduces the intensity of the characteristic peaks. Because the beam-sample interaction volume is proportional to the accelerating potential of the electrons, the accelerating potential that is too high degrades the lateral resolution due to the increase in secondary electron generation. The electron beam current is

usually selected to produce a suitable X-ray count rate of around 3000 counts per second [3]. If the beam current is too low, the collected spectrum will be statistically unsafe. If the beam current is too high, it tends to increase the artifacts, such as the sum peaks and Si escape peaks.

Once the SEM parameters are properly adjusted, two geometric parameters may be adjusted to improve the collector efficiency. These are the solid angle (SA) and the take-off angle (TOA). As shown in Figure 4, it is desirable to have the SA relatively high since a larger SA corresponds to more X-rays detected. The high SA can be achieved by reducing the working distance of the SEM. The TOA is shown in Figure 5, where a TOA of 35° or greater is recommended in order to increase the collector efficiency. This is because the intensity of X-rays is dependent on the angle at which they leave the sample.

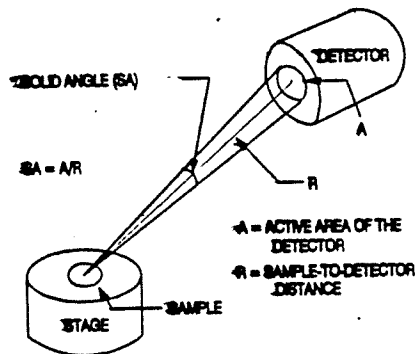


Figure 4: SA.

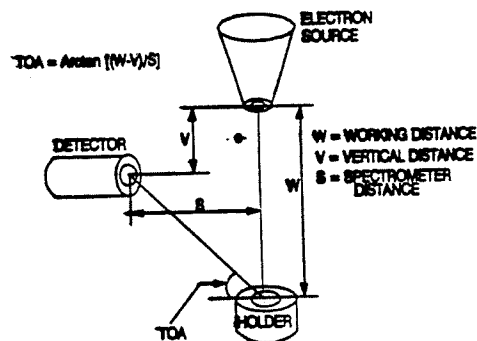


Figure 5: TOA.

In this project, qualitative EDS analysis was explored to lay a groundwork for future studies utilizing the Kevex X-ray EDS system at R. I. T.

EXPERIMENT

Three samples were prepared for EDS qualitative analysis: Al on Si, Cu on Si, and a PZT ferroelectric film on Si. Approximately 0.4 μm of Al was thermally evaporated on a Si wafer using resistive heating and patterned into lines and spaces. Similarly, approximately 1.7 μm of Cu was deposited on a Si wafer, but not patterned. The PZT film was prepared by a Sol-gel process and spun onto a Si wafer to form approximately 0.1 micron film.

A Link X-ray EDS system attached to a Cambridge SEM was used to perform the EDS qualitative analysis on the three samples described above. A PGT X-ray EDS system attached to a Hitachi S-4000 SEM was also utilized to perform some of the more advanced EDS qualitative analysis, namely X-ray mapping, using an alloy material that was specially prepared for such analysis.

RESULTS/DISCUSSION

Figure 6 is the X-ray spectrum obtained from the Al on Si sample. As expected, this X-ray spectrum contains the Al and Si characteristic peaks. Also, notice that a small amount of S and Cl was detected. It is possible that these contaminants are from the etching solution used to pattern the Al layer, and this should be further investigated. Figure 7 is the X-ray spectrum of the Cu on Si sample, and it clearly illustrates the Cu characteristic peaks. However, notice that although the Cu layer was not patterned, the underlying Si substrate is detected. This is due to the scattering effect where electrons penetrated all the way through the Cu layer and reached the Si substrate. Figure 8 is the X-ray spectrum of the PZT ferroelectric film, which displays the Pb, Zr, and Ti characteristic peaks. Again, notice that the spectrum contains a Si peak due to the scattering effect. However, the intensity of this Si peak is much higher than that of the Si peak from the Cu-Si sample spectrum, because the PZT film is only about 0.1 micron thick (as oppose to 1.0 micron of Cu).

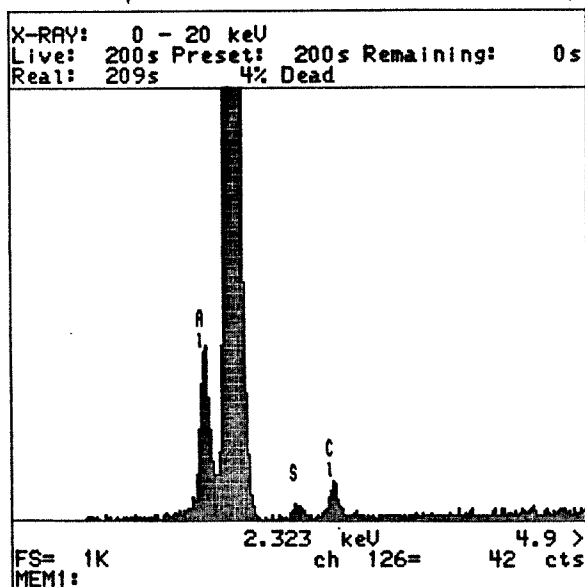


Figure 6: Patterned Al on Si.

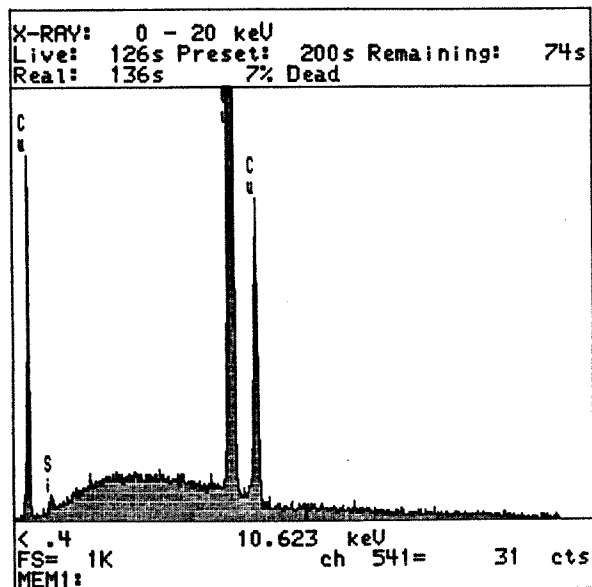


Figure 7: Cu on Si.

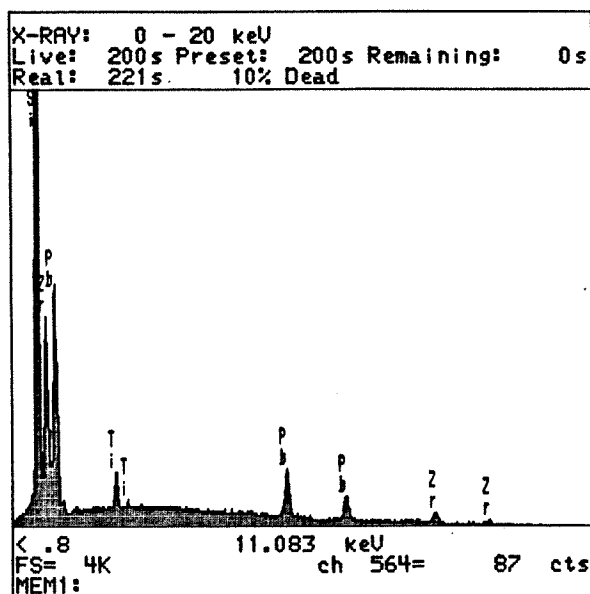


Figure 8: PZT.

Figure 9 is the X-ray mapping obtained from a special alloy. It clearly displays the spatial distribution of all the elements present in this alloy.

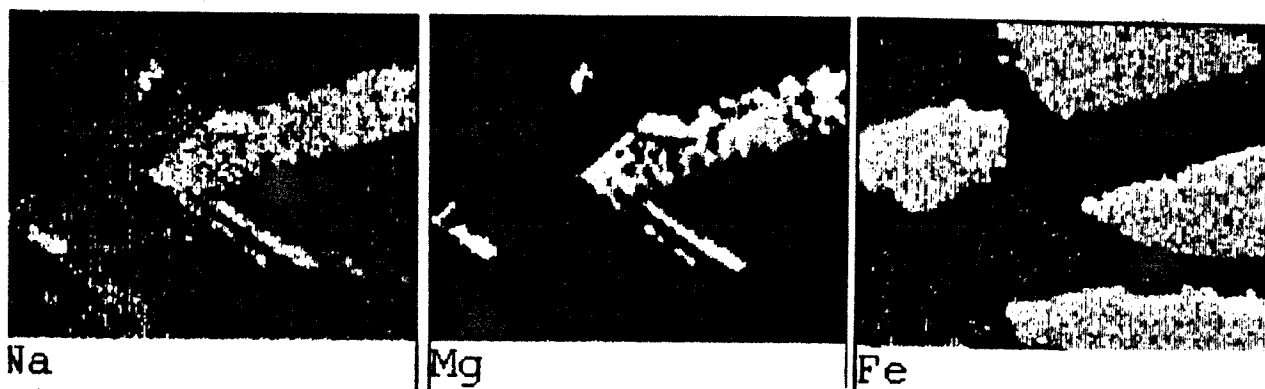


Figure 9: X-ray mapping.

CONCLUSION

An overview of EDS qualitative X-ray microanalysis was presented with emphasis on some of the artifacts present in an EDS system, such as Bremsstrahlung, sum peaks, and Si escape peaks. Also, some of the system parameters, such as the SA, TOA, electron acceleration potential, and beam current, were discussed in order to reduce the artifacts and to obtain better results. By obtaining the X-ray spectra of a variety of samples, the qualitative elemental identification capability of an EDS system was demonstrated. Some of the more advanced qualitative analysis, such as X-ray mapping, was also demonstrated using a special alloy.

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