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A Guideline for Increasing Efficiency of TEM/EDS Data Collection by Dwell Time Optimization

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A Guideline for Increasing Efficiency of TEM/EDS Data Collection by Dwell Time Optimization

Abstract

Composition analysis using energy dispersive spectroscopy (EDS) mapping in transmission electron microscopy (TEM) is crucial in the semiconductor industry for development of new products and enhancement of production yields. High quality EDS data relies on acquiring sufficient X-ray counts from the TEM sample. The amount of time that the electron beam interacts with the sample generating X-rays per pixel within the mapping area is known as dwell time, which is an EDS system parameter that governs optimum data acquisition. However, a systematic study to optimize this parameter has not been previously reported. An analytical expression was derived that enabled the prediction of a dwell time range that optimizes the total X-ray signal collected during the EDS data collection. Experimental results from multiple materials across several TEM/EDS systems confirmed the validity of the expression. The results of this study provide a guideline for increasing efficiency of TEM/EDS data collection from different materials using a variety of TEM/EDS systems through the optimization of EDS dwell time.

A Guideline for the Optimization of Dwell Time to Achieve Better Efficiency in TEM/EDS Data Collection

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Introduction

Background

- Elemental composition analysis using transmission electron microscopy (TEM) / energy dispersive spectroscopy (EDS) mapping is crucial in the semiconductor industry to solve semiconductor processing problems that occur on the nm scale.
- Point analysis, elemental line profile, and/or elemental distribution can be extracted from EDS maps.

Challenges to Collecting High Quality EDS Data

- High TEM sample stage drift rates compromise quality of data collected.
- Long electron beam exposure times cause significant structural degradation to electron beam sensitive materials in TEM samples.

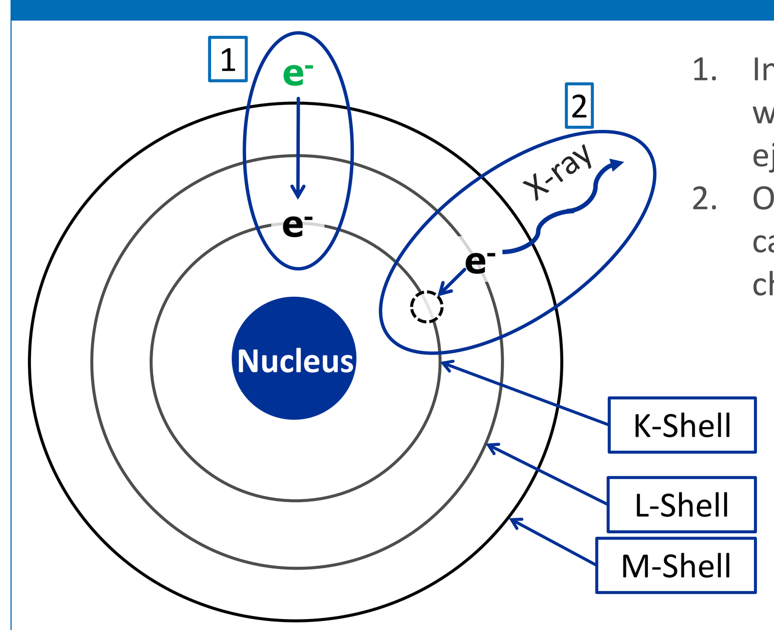
Solutions to these Challenges

- Factors such as beam current, TEM sample thickness, process time, dwell time and total data collection time affect the quality of EDS data.
- Dwell time, the time allowed for EDS signal generated by the electron beam for each pixel, has a direct impact to overcome these challenges.

Goal

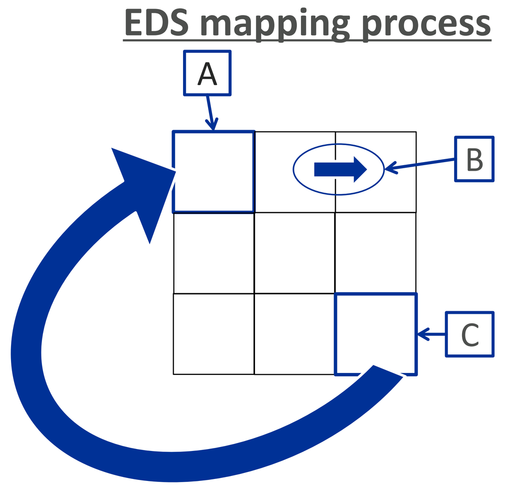
- Determine the total X-ray counts as a function of dwell time in a given time theoretically and experimentally, and provide a guideline for increasing the efficiency of TEM/EDS data collection through the optimization of EDS dwell time.

EDS Introduction



1. Incident electron from electron beam interacts with inner shell electron of elemental atom, ejecting it creating a vacancy in the shell.
2. Outer shell electron moves into this vacancy, causing a release of energy in the form of a characteristic X-ray.

EDS mapping process



A. Each Square represents a pixel in the EDS mapping area
B. Pixel to pixel transition time
C. EDS map frame ends, returns for next frame (time returning to beginning is fly-back time)

Theoretical Analysis

- Analyzing the EDS mapping process created a derivation of an expression for the total X-ray counts as a function of the dwell time shown below:

$$S = (CPS(1 - DT)) * T_t * \left(1 - \frac{nT_p + T_f}{n\tau_D + nT_p + T_f}\right) \qquad (1)$$

- S = Total X-ray counts collected – Dependent variable under consideration
- CPS = Counts per second generated from the sample
- DT = Dead time of the system during X-ray counts/signal acquisition
- T_t = Total data collection time
- τ_D = Dwell time – Independent variable under consideration
- T_p = Pixel to pixel transition time
- T_f = Fly-back time for each frame
- n = Number of pixels in the scan area

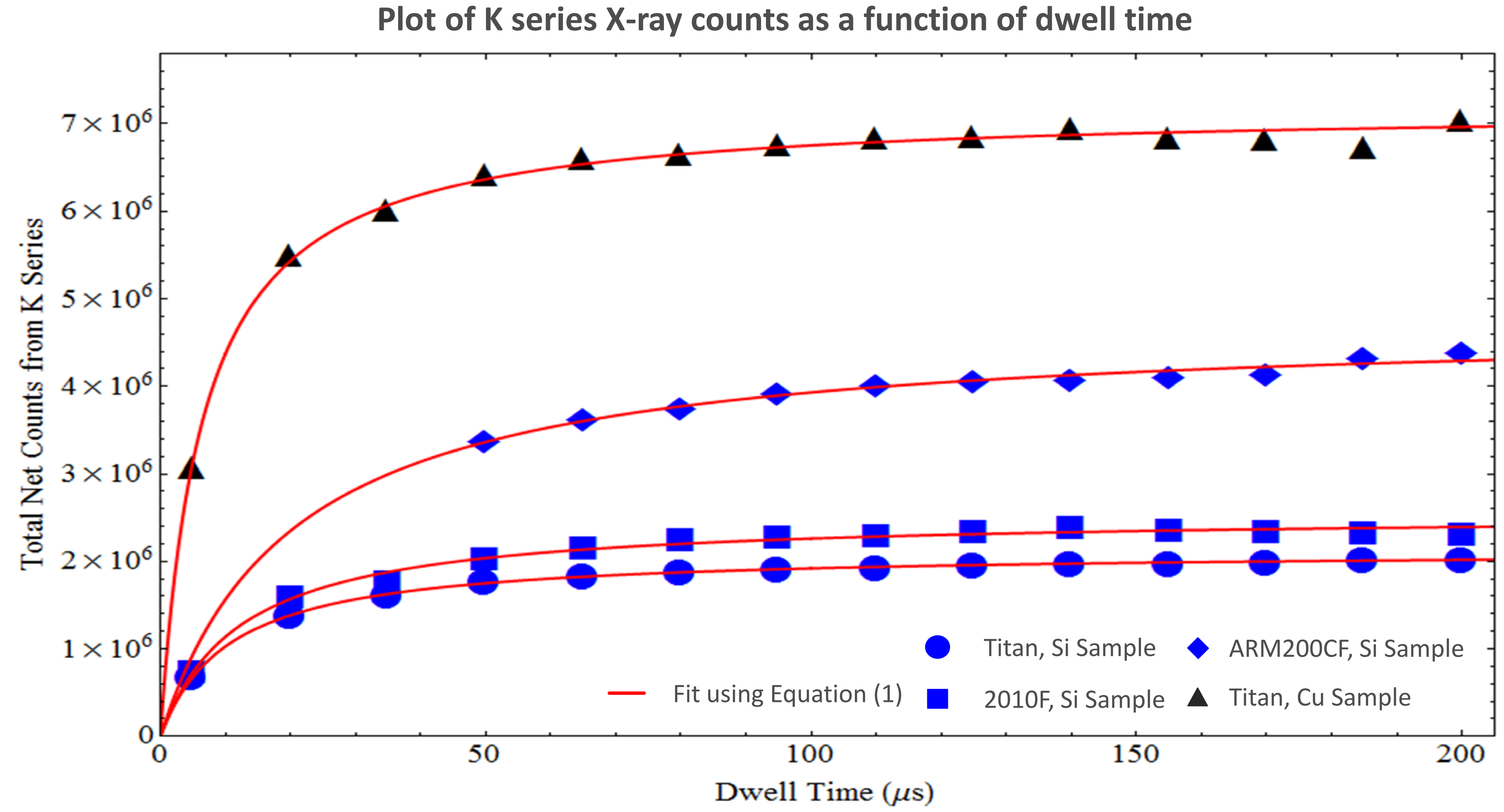
Experimental Design

Experiment	Purpose	Material
I	Determination of relationship between total X-ray counts as a function of dwell time using Titan	Si, Cu
II	Confirm the relationship on similar TEM, Titan versus 2010F, with the same EDS system	Si
III	Confirm the relationship on different TEM/EDS system, Titan and 2010F versus ARM200CF	Si

- TEMs set to a specimen current of 2.2 nA ± 10%, measured in-situ.
- Total EDS data collection time (T_t) for each data point was 600 seconds.
- Spectrum process times set to 3.0 µs for the Aztec systems and 3.2 µs for the NS7 system.
- Sample thicknesses were measured using electron energy loss spectroscopy (EELS) pre and post data collection to ensure no significant change in sample thickness.

Results

Plot of K series X-ray counts as a function of dwell time



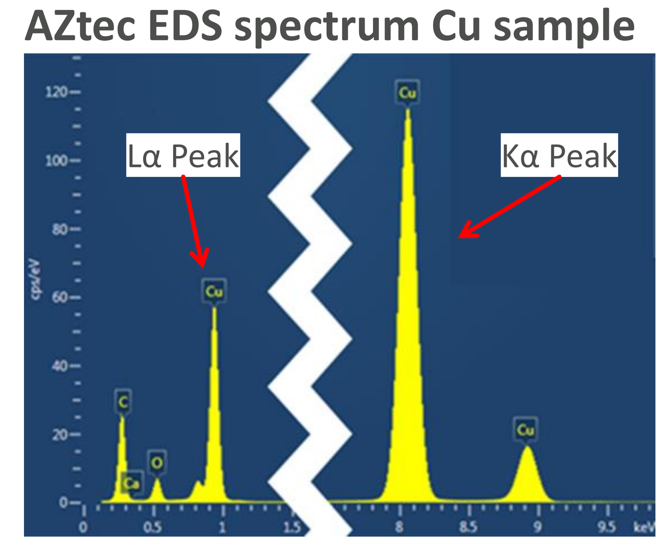
Experimental hardware specifications

TEM	Electron Source	EDS Detector / Software	Solid Angle (sr)
Titan	Shottkey FEG	X-MaxN 80mm^2 Windowless SDD / AZtec	0.17
2010F	Shottkey FEG	X-MaxN 80mm^2 Windowless SDD / AZtec	0.16
ARM200CF	Cold FEG	Centurio 100mm^2 Windowless SDD / NS7	0.86

Effects of dwell time on the collection of X-ray signal with different energies

- Experiments confirmed that the collection of X-ray signal with different energies is independent of dwell time setting.

AZtec EDS spectrum Cu sample



Dwell Time (µs)	Cu Lα Peak Counts	Cu Kα Peak Counts	Lα /Kα Ratio
5	57088	116800	0.49
95	95275	193808	0.49
200	130081	265153	0.49

Discussion

Experiment I

- X-ray counts from the Cu sample are significantly higher than those from the Si sample due to higher X-ray generation rate from Cu.

Experiment II

- The number of X-ray counts collected from the Titan system matched closely to the counts collected from the 2010F system, due to the similarity of the systems.

Experiment III

- The number of X-ray counts collected from the ARM200CF system are higher than the counts collected from either of the other two systems, which is expected due to the large difference in solid angle between the two types of EDS detectors.

Data Analysis

- All experimental data sets are in good agreement with the derived analytical expression with R^2 values greater than 0.99.

Detector Dwell Time versus X-ray Signal Relationship

- The Cu sample data from Experiment I confirmed the total X-ray counts collected were not more sensitive at any particular X-ray energy due to change of dwell time, shown by Cu Lα/Kα ratios being constant.

Sample Thickness

- EELS analysis confirmed that the thickness of the sample did not significantly change during the duration of Experiment III, eliminating sample thickness as a possible reason for trends in total X-ray counts.
- Verified using Log – Ratio method [1]
- Pre EDS data collection was 63 nm +/- 4 nm
- Post EDS data collection was 67 nm +/- 4 nm

Conclusion & Deliverable

- High Quality EDS maps can be collected by the optimization of dwell time setting based on different TEM/EDS systems and materials in this study.
- A guideline, that is applicable to almost all TEM/EDS systems and any material, for the selection of dwell time was developed.

Future Work

- Analysis of the relationship between EDS system process time and the quality of EDS map data.
- Investigation into the relationship between sample X-ray generation and use of different electron sources.

Acknowledgments

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Reference

[1] Egerton R. *Electron Energy-Loss Spectroscopy In The Electron Microscope*. New York: Springer; 2011.