EDS

Analysis of the elemental composition and thickness of a Fe-Ni film on Si using Bruker ESPRIT and SAMx STRATAGem

Application Note # EDS-11

Introduction

Quantitative analysis of a bulk sample requires that the composition of the sample is homogeneous over the analyzed volume. For inhomogenous samples the calculation of the matrix effects is not correct and this can lead to wrong results in the element concentrations. For samples containing a layer structure a different quantitative evaluation has to be applied. This can be provided with the standard-based analysis in ESPRIT in combination with the STRATAGem software.

Therefore, the quantitative analysis of a Fe-Ni film on a Si substrate was performed using ESPRIT and STRATAGem. The sample analyzed was provided by the German Federal Institute for Materials Research and Testing (BAM) and the Korean Research Institute of Standards and Science (KRISS). It consists of a 200 nm thick Fe-Ni film that has been sputtered on a Si wafer, as shown in Figure 1.

The elemental composition of Fe-Ni alloy was certified by ICP-MS to be

- Fe: 50.02 ± 1.23 at.%
- Ni: 49.98 ± 1.23 at.%

Experiment and results

Spectra of this sample and of pure Si, Fe and Ni standards were acquired for a standards database. The spectra were acquired with acceleration voltages of 12, 15, 20, 25 and 30 kV.

Figure 1
Illustration of the analyzed Fe-Ni layer on Si.

For sufficient spectrum statistics, acquisition times were 60 s per spectrum and input count rates were between 10-30 kcps depending on the accelerating voltage. This is important because the interaction volume changes with the acceleration voltage and the peak intensities of Fe, Ni and Si provide information not only about the composition of the film but also about the film thickness on the substrate. Figure 2 shows a Monte Carlo simulation of the interaction volumes in the layer and substrate for 12, 15, 20, 25, 30 kV.

The STRATAGem software tool can be used to calculate film composition and thickness with the help of the K ratios. K ratios are the ratios of the peak intensities of the sample and the standard normalized to beam current and live time,

$$K = \frac{I_{FE\ K\alpha,\ sample}}{I_{FE\ K\alpha,\ std.}}$$

e.g. for a pure element standard: $0 \leq K \leq 1$.

The K ratios for Si, Fe and Ni were analyzed using ESPRIT's HSQuant module and found to be 0.154, 0.414 and 0.438 respectively for the 12 kV spectrum, as shown in Figure 3.

a)

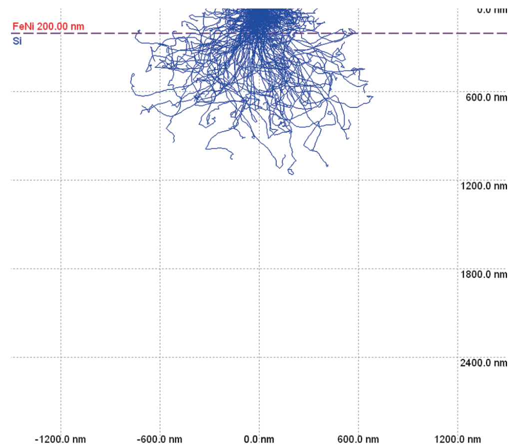
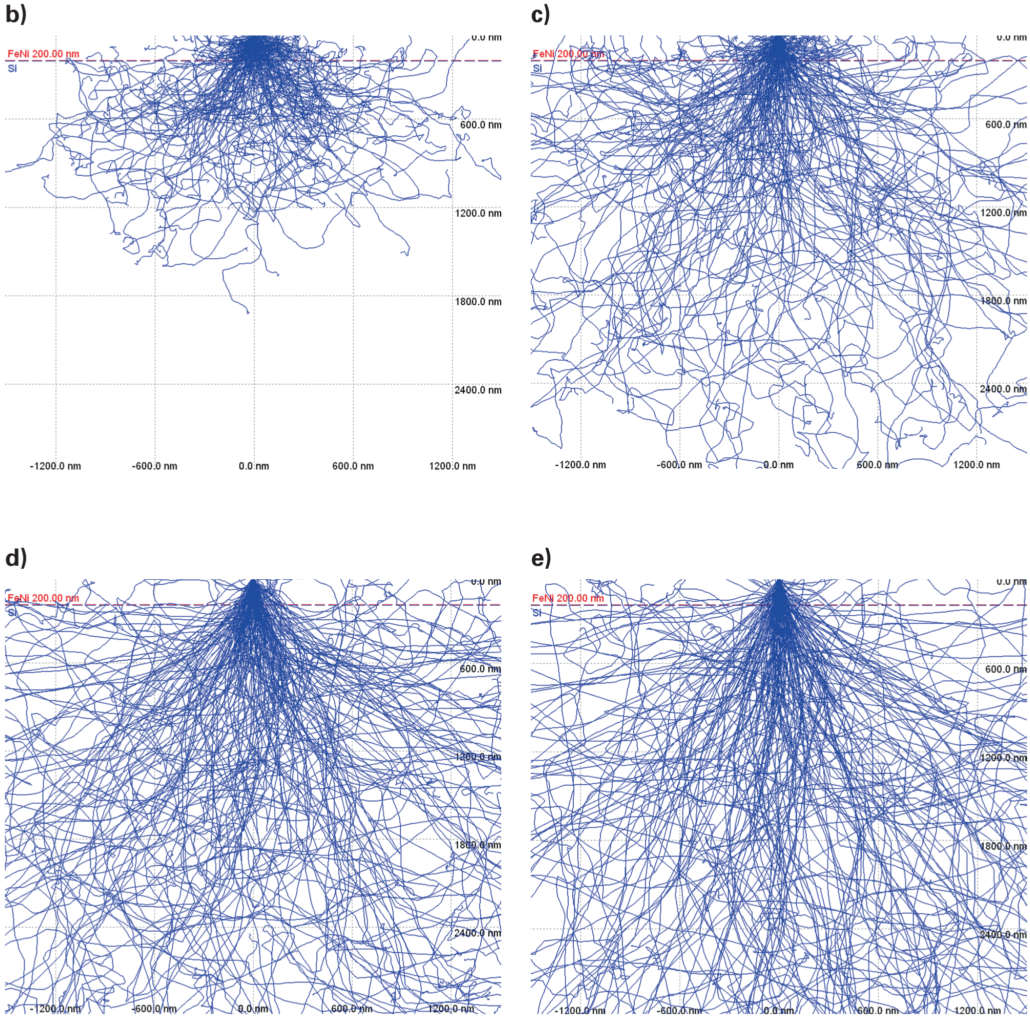


Figure 2
Monte Carlo simulations of electron beams hitting the sample with acceleration voltages of 12 kV (a), 15 kV (b), 20 kV (c), 25 kV (d) and 30 kV (e).

b) c) d) e)



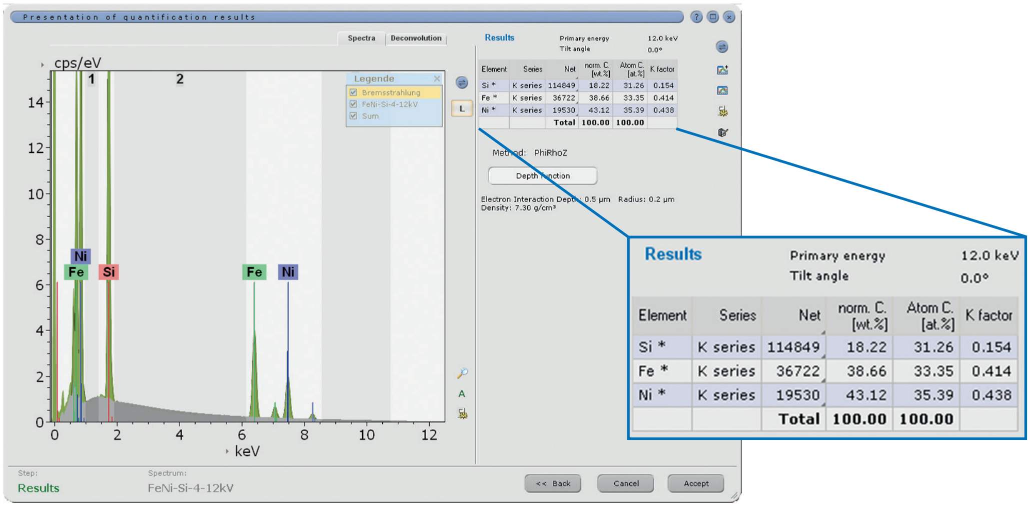


Figure 3
ESPRIT quantification results, including K ratios (K factors)

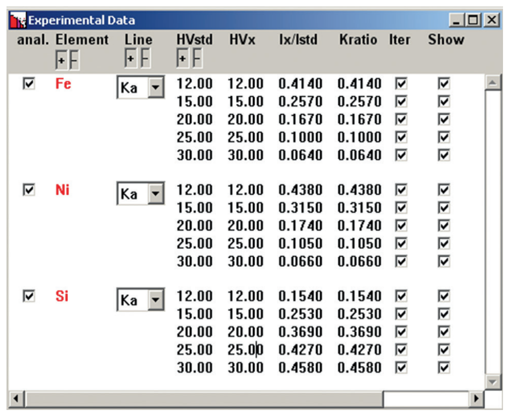
The elemental concentrations have no meaning because the sample is not homogeneous but the K ratios are used for further analysis.

Since the center of the interaction volume moves from the Fe-Ni film for low HV to the Si substrate for higher HV, the K ratio of Si will increase and the K ratios of Fe and Ni will decrease with increasing HV. All K ratios are transferred into the Stratagem software. The STRATAGem window shows the K ratios for the sample ("Kratio"), the peak intensity ratio of the sample to the standard ("Ix/Istd"), the accelerating voltage used for the sample ("HVx") and for the standard ("HVstd"), as depicted in Figure 4.

A description of the analyzed sample, consisting of substrate and layer definitions, has to be provided for STRATAGem (Figure 5). Red numbers indicate fit variables, the values of which will be fitted according to the experimental K ratios (thickness, layer composition, substrate composition).

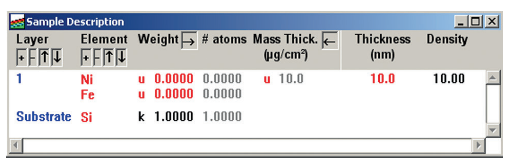
With these data theoretical K ratios are fitted to the experimental ones, as displayed in Figure 6. Film composition and thickness are optimized based on the fit results.

For the calculation of the film thickness a density has to be provided. This was set to the theoretical density of a bulk FeNi sample. As this density can be different for thin FeNi films this may also affect the determined film thickness.


Experimental Data

anal.	Element	Line	HVstd	HVx	Ix/Istd	Kratio	Iter	Show
[x]	Fe	Ka	12.00	12.00	0.4140	0.4140	[x]	[x]
			15.00	15.00	0.2570	0.2570	[x]	[x]
			20.00	20.00	0.1670	0.1670	[x]	[x]
			25.00	25.00	0.1000	0.1000	[x]	[x]
			30.00	30.00	0.0640	0.0640	[x]	[x]
[x]	Ni	Ka	12.00	12.00	0.4380	0.4380	[x]	[x]
			15.00	15.00	0.3150	0.3150	[x]	[x]
			20.00	20.00	0.1740	0.1740	[x]	[x]
			25.00	25.00	0.1050	0.1050	[x]	[x]
			30.00	30.00	0.0660	0.0660	[x]	[x]
[x]	Si	Ka	12.00	12.00	0.1540	0.1540	[x]	[x]
			15.00	15.00	0.2530	0.2530	[x]	[x]
			20.00	20.00	0.3690	0.3690	[x]	[x]
			25.00	25.00	0.4270	0.4270	[x]	[x]
			30.00	30.00	0.4580	0.4580	[x]	[x]

Figure 4
All experimental data as entered into STRATAGem


Sample Description

Layer	Element	Weight	# atoms	Mass Thick. (µg/cm²)	Thickness (nm)	Density
1	Ni	u 0.0000	0.0000	u 10.0	10.0	10.00
	Fe	u 0.0000	0.0000			
Substrate	Si	k 1.0000	1.0000			

Figure 5
STRATAGem's layer description dialog, variables that will be fitted are displayed in red

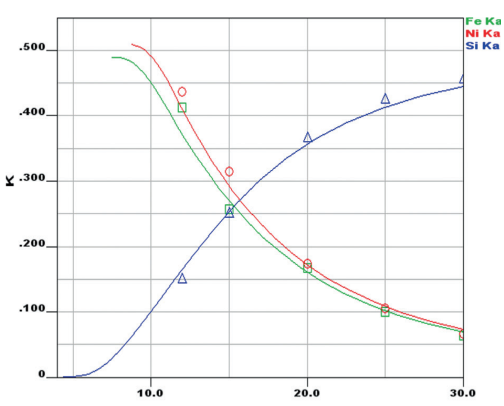


Figure 6
K ratio vs. HV plot with fit curves

The resulting fit parameters can be seen in Figure 7. They show a good agreement in composition (Fe 50.3 at%) and film thickness (185 nm) when compared to the provided certified values.

Layer	Element	Weight	# atoms	Mass Thick. (µg/cm²)	Thickness (nm)	Density
1	Ni	u 0.5093	0.4968	u 155.1	184.6	8.40
	Fe	u 0.4907	0.5032			
Substrate	Si	k 1.0000	1.0000			

Figure 7
Fit results, displaying layer composition and thickness close to the expected values.

Summary

The advantage of the use of STRATAGem in combination with Bruker ESPRIT is:

- A wide range of variables can be set to fixed values or used as fit parameters. Result graphs can be directly checked f or agreement,
- The open model provides a flexible setup of the sample structure,
- Graphs of K ratios vs. HV or K ratios vs. thickness can be generated.

This method is good for the analysis of layers with thicknesses ranging from some nm to a few µm, depending on HV and type of layer. It also provides accurate results for multilayer structures.

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