Analysis of Elemental Composition of Fe_{1-x}Ni_{x} and Si_{1-x}Ge_{x} Alloy Thin Films by EPMA and µ-XRF

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It was demonstrated in the past that electron probe microanalysis (EPMA) can be applied to determine accurately both elemental composition and thickness of thin films [1-3] by using the dedicated software package for thin film analysis Stratagem [4]. A relatively small number of film materials such as pure metallic films of platinum and nickel [1], binary alloys of Fe-Ni [2], and Pt-Ni-Co ternary alloy films [3] has been reported in literature as working successfully. Further, the software can be applied ‘inversely’, i.e. by feeding it with the thickness of the film and using the determined mass coating (or mass deposition, or mass thickness, in µg/cm²), one can easily calculate the film density, which for porous layers leads us to the true film porosity [5].

The present study repeats measurements on an already tested system of Fe-Ni thin films on silicon [2] and reports for the first-time analysis results of analysis on Si-Ge thin films deposited on a non-conductive aluminum oxide substrate. Standard based and standardless EPMA (with EDS) results were used in combination with Stratagem for the quantification.

X-ray fluorescence analysis (XRF) can be used for the determination of elemental composition and thickness of such films as well. In this case XRF with a micro-focus X-ray source (µ-XRF) attached to an SEM was applied. For quantification a fundamental parameter (FP) approach has been used to calculate standard-based and standardless results [6]. Compared to EPMA, XRF has a larger information depth and a higher elemental sensitivity because of a generally lower background.

Both thin film systems have been chosen as samples of an international round robin test (RRT) organized in the frame of standardisation technical committee ISO/TC 201 ‘Surface chemical analysis’ under the lead of KRISS. The main objective of the RRT is to compare the results of atomic fractions of Fe_{1-x}Ni_{x} and Si_{1-x}Ge_{x} alloy films obtained by different surface analysis techniques, such as X-ray Photoelectron Spectroscopy (XPS), Auger Electron Spectroscopy (AES) and Secondary Ion Mass Spectrometry (SIMS) applied in the depth-profiling operation mode [6].

Five samples of different atomic fractions of each thin film system, i.e. Fe_{1-x}Ni_{x} and Si_{1-x}Ge_{x}, have been grown by ion beam sputter deposition on silicon and Al2O3 wafers, respectively. Reference FeNi and SiGe films with well-known elemental composition and thickness have been also supplied for standard based analysis. The atomic fractions of all the samples including the references have been certified by RBS (Rutherford Backscattering Spectrometry) and ICP-AES (Inductively coupled plasma atomic emission spectroscopy) [7].
The results of the standard-based and standardless EPMA/Stratagem and \( \mu \)-XRF analyses are shown in Figure 1. Note the good agreement (linearity) of all data with the reference values.

A detailed report including the data obtained by all the RRT participants using the other, depth profiling techniques as specified above will be published soon.

References:


**Figure 1.** Results of standard-based and standardless thin film analyses by EPMA/Stratagem (Lab 1, Lab 2) and \( \mu \)-XRF (Lab 3) for the FeNi and SiGe films (in the ordinate) represented as molar ratio of Fe (for FeNi layers deposited on silicon wafer) in dependence on the reference values of Fe measured by ICP-AES (left panel), and same representation of Si molar ratio (for SiGe layers deposited on a non-conductive aluminum oxide substrate) relative to the reference values as measured by RBS.