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Measurement of Elemental Composition of FeNi and SiGe Thin Films by Electron Probe Microanalysis with Stratagem Software

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It was demonstrated in the past that the electron probe microanalysis (EPMA) can be applied to determine accurately both elemental composition and thickness of thin films in a non-destructive manner [1-3] by using the dedicated software package for thin film analysis Stratagem [4]. A relative 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, 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 and reports - for the first-time – results of analysis on Si-Ge thin films deposited on a non-conductive aluminium oxide substrate. Both thin film systems have been chosen as samples of an international round robin test (RRT) organized in the frame of ISO technical committee ISO/TC 201 'Surface chemical analysis' (https://www.iso.org/committee/54618.html) under the lead of KRISS, Korea. The main objective of the RRT is to compare the results of atomic fractions of $Si_{1-x}Ge_x$ and $Fe_{1-x}Ni_x$ alloy films obtained by different surface analysis techniques, such 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 Al_2O_3 wafers, respectively. Reference FeNi and SiGe films with well-known elemental composition and thickness have been also supplied. The atomic fractions of all the samples including the references have been certified by RBS and ICP-AES [6]. The results of the EPMA/Stratagem analysis are shown in Figure 1. Note the excellent agreement (linearity) of all data with the certified values.

If the very good EPMA/Stratagem results for the FeNi system were expected due to previous studies [2], the data obtained for the SiGe films are particularly valuable, because of the challenging insulator substrate of Al_2O_3. The conductivity of the $Si_{1-x}Ge_x$ surface necessary for charging-free analysis was ensured by applying conductive copper tape onto film surface down to sample stage. Four accelerating voltages, 15, 20, 25 and 30 kV, have been applied, so that the Ge Kα X-ray line at 9.87 keV could be excited. Each sample has been measured at five locations to ensure good statistics. The beam current has been measured with a calibrated external Picoammeter. As for the Al_2O_3 substrate, only the Al Kα line has been included in the analysis, the existent stoichiometry being a-priori defined in Stratagem. Further analysis details can be seen in Figure 2, where a screen shot of the Stratagem 'Sample description' window corresponding to the $Si_{0.2}Ge_{0.8}$ thin film sample is illustrated. It should also be noted that the

density of the 'unknown' layer has been assessed from the literature data for densities of pure Si and Ge and from the nominal atomic fractions.

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

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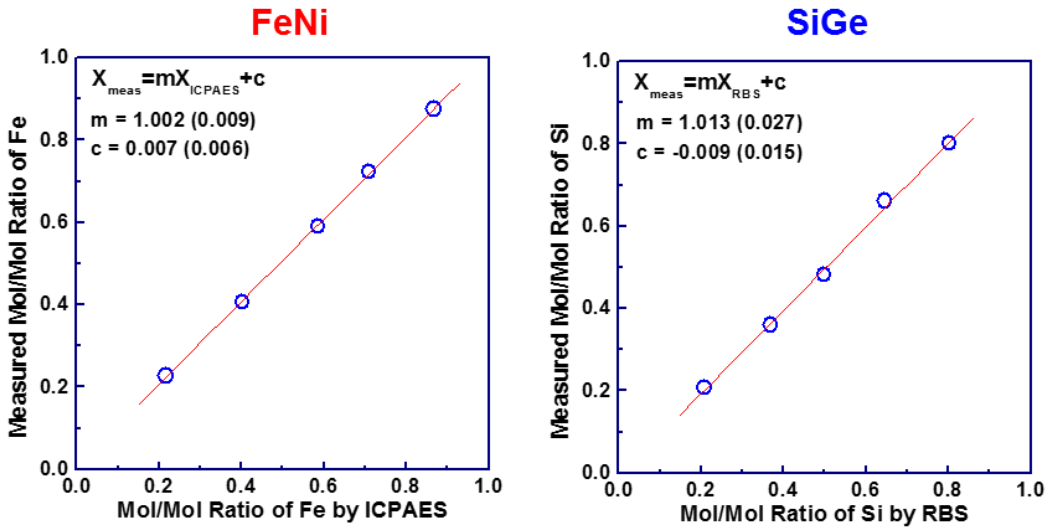

Figure 1. Results of thin film analysis by EPMA/Stratagem for the FeNi and SiGe films (in the ordinate) represented in dependence on the reference values of Fe (for FeNi layers) and Si (for SiGe layers) measured by ICP-OES and RBS, respectively.

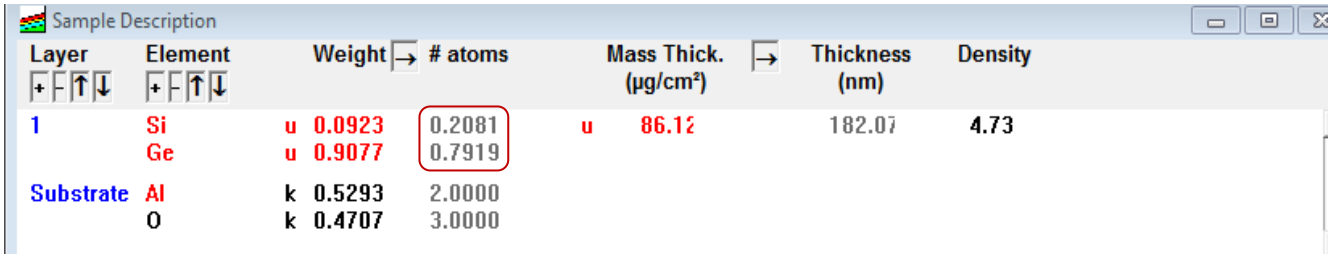

Figure 2. Excerpt from Stratagem with the results of the analysis of the (nominal) $Si_{0.2}Ge_{0.8}$ thin film sample with a given (theoretical) density of 4.73 g/cm^3.

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