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Boron Characterization in Intermetallic Bulk or Thin Film Alloys using Scanning Electron Microscopy and Electron Probe Microanalysis

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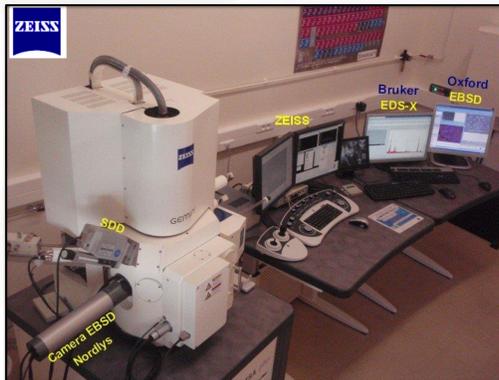


Scanning Electron Microscopy Facilities and Electron Probe Micro-Analyser



JEOL Scanning Electron Microscope - JEOL 840A

SAMx Energy Dispersive X-ray Spectroscopy (EDX - Si - Li)

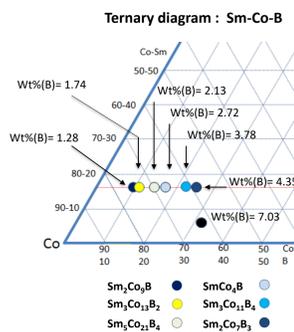
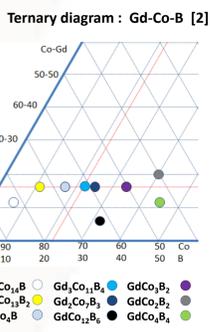
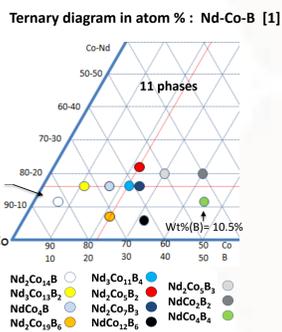


Field Emission Gun Scanning Electron Microscope (FESEM) - ZEISS ULTRA+, Bruker Energy Dispersive X-ray Spectrometry (SDD), Electron BackScatter Diffraction (EBSD)



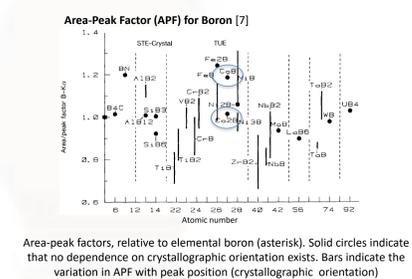
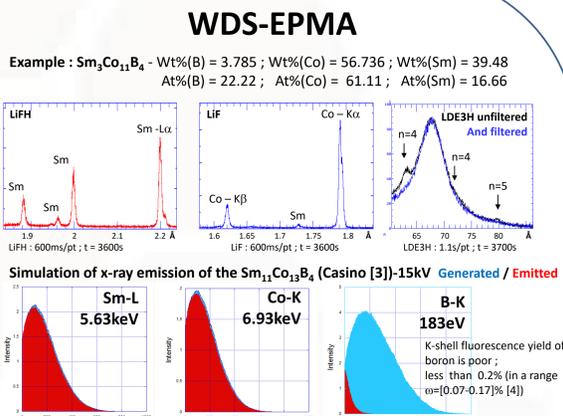
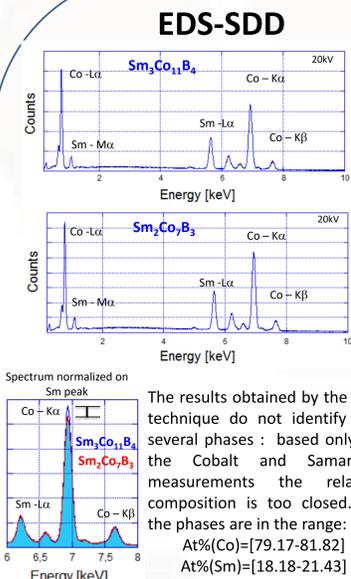
Electron Probe Micro-Analyser (EPMA - JEOL 8800) Equipment: 5 wavelength dispersive x-ray spectrometers (WDS) each with two crystals
1: PETH / LDE3H 2: TAP / LDE1H 3: LED2 / LDE2H
4: PETJ / LiFH 5: PETH / LiF (Xe)

Sample preparation and analysis of bulk RE-M-B (RE=Nd, Gd, Sm) & (M=Fe,Co)



Several Sm-Co-B samples were prepared by induction melting in a pure argon gas atmosphere. Sm-Co-B ingots were sealed in a quartz tube and annealed for three weeks at 900 ° C. RE-M-B alloys are very sensitive to oxidation, which complicates sample preparation for microscopy. Indeed, it was very challenging to carry out efficient preparation without creating damage or causing oxidation. Water-free lubricant is preferable, so absolute ethanol and glycol were used. To limit oxidation, samples were prepared just prior to observation and transport in a vacuum desiccator. Polishing was carried out using grinding paper and then polishing cloth with 0.25 µm alumina or diamond particles.

Results on bulk Sm-Co-B alloys



Results at 15kV - B (LDE3H) - Sm (LiFH) - Co (LiF); no cold finger, i # 50 nA

Weight percent	Co	Sm	Total	Atomic ratio	Co	Sm	Total
1	56.824	39.866	100.000	1.438	62.773	17.253	80.026
2	56.324	39.866	100.000	1.438	62.773	17.253	80.026
3	56.324	39.866	100.000	1.438	62.773	17.253	80.026
4	56.324	39.866	100.000	1.438	62.773	17.253	80.026
5	56.324	39.866	100.000	1.438	62.773	17.253	80.026
Minimum	4.021	36.394	100.106	Minimum	23.2996	59.7647	16.3437
Maximum	4.155	39.394	100.836	Maximum	23.8061	60.7929	16.5467
Average	4.10	36.74	100.46	Average	23.64	59.95	16.41

Results at 15kV - B (by difference; off-line calculation) - Sm (LiFH) - Co (LiF); no cold finger, i # 50 nA

Weight percent	Co	Sm	Total	Atomic ratio	Co	Sm	Total
1	56.824	39.866	100.000	1.438	62.773	17.253	80.026
2	56.324	39.866	100.000	1.438	62.773	17.253	80.026
3	56.324	39.866	100.000	1.438	62.773	17.253	80.026
4	56.324	39.866	100.000	1.438	62.773	17.253	80.026
5	56.324	39.866	100.000	1.438	62.773	17.253	80.026
Minimum	3.316	36.394	100.394	Minimum	19.9684	60.1974	16.5467
Maximum	3.985	36.394	100.764	Maximum	23.2018	62.7733	17.2583
Average	3.65	36.74	99.62	Average	21.56	61.38	16.87

Results in two steps : 15kV - Sm (LiFH) - Co (LiF); 5kV - B (LDE3H); cold finger, i # 50 nA

Weight percent	Co	Sm	Total	Atomic ratio	Co	Sm	Total
1	56.824	39.866	100.000	1.438	62.773	17.253	80.026
2	56.324	39.866	100.000	1.438	62.773	17.253	80.026
3	56.324	39.866	100.000	1.438	62.773	17.253	80.026
4	56.324	39.866	100.000	1.438	62.773	17.253	80.026
5	56.324	39.866	100.000	1.438	62.773	17.253	80.026
Minimum	3.65	36.394	100.394	Minimum	21.654	16.714	61.303
Maximum	3.74	39.96	101.39	Maximum	21.983	16.714	61.303
Average	3.71	39.93	100.74	Average	21.885	16.725	61.343

The values obtained at 15kV for Sm₂Co₇B₃ & Sm₂Co₁₁B₄ phases are dispersed in a range about wt%=0.2 and wt%= 0.5. No changes have occurred despite the two steps calculation. It could be due to several things: difference between the shape of the Boron peak of the standard and the sample, the primaries phases Sm₂Co₇B₃ & Sm₂Co₁₁B₄ could be unstable and split into two phases (the average of the results in the Sm₂Co₁₁B₄ stayed at the expected value) or could not exist (Sm₂Co₇B₃) ! For others phases the computation focus to 0.1%

Thin films analysis based on EDX and WDX measurements

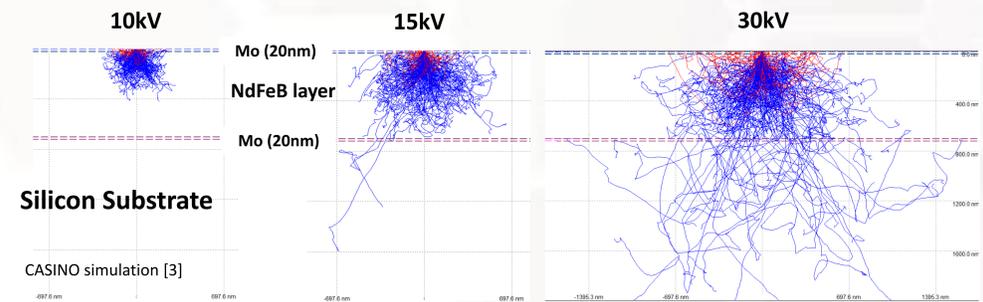
Hard magnetic films have many potential applications in micro-systems and serve as model systems to study the physics of magnetisation reversal in these technologically important materials. The functional properties of hard magnetic materials, in particular their resistance to demagnetisation (i.e. their coercivity), depend critically on their microstructure.

Films of NdFeB were made by high rate triode sputtering [5]. Buffer and capping layers such as Ta are used to prevent interdiffusion into the substrate and oxidation from the atmosphere, respectively. Films were characterised by cross-sectional SEM imaging coupled with Energy or Wavelength Dispersive Spectrometries (EDX & WDX). Boron content was determined with EPMA measurements (LDE3H), Nd and Fe with EDX and PETH or LiF crystals.

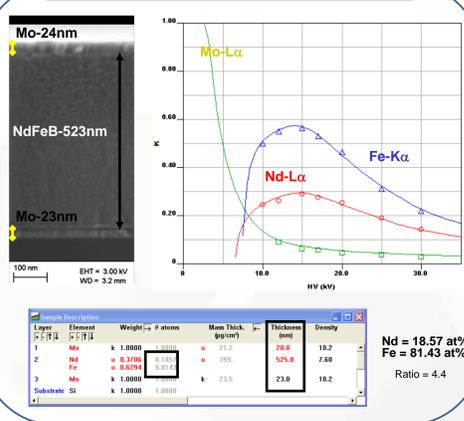
Changing the energy of the incident electrons varies the volume of the electron-matter interaction in the sample : at low beam voltage for example, the upper layers are favoured compared to buried layers and the substrate. A multi-layer sample is characterized by doing x-ray analyses at different beam voltages. The results are treated by off-line calculation software packages (STRATAGEM / SAMx [6]). The calculations use the nominal layer architecture as starting input, estimating fluorescence and absorption for the given geometry. At the end of an iteration process, curves of predicted relative intensities corresponding to the chemical compositions and thicknesses found are drawn and compared with the experimental data to validate the analysis.

For these experiments, Mo capping layers were used since the mass absorption coefficient of the K α line (183eV) of Boron by Ta is very high. The half-value layer for B absorption by Ta and Mo is 20nm (M.A.C.= 20,820 cm²g⁻¹) and 130nm (M.A.C.= 5,238 cm²g⁻¹), respectively. The absorption for Fe-K α (6.4keV) and Nd-L α (5.23keV) are weak (M.A.C. less than 500 cm²g⁻¹).

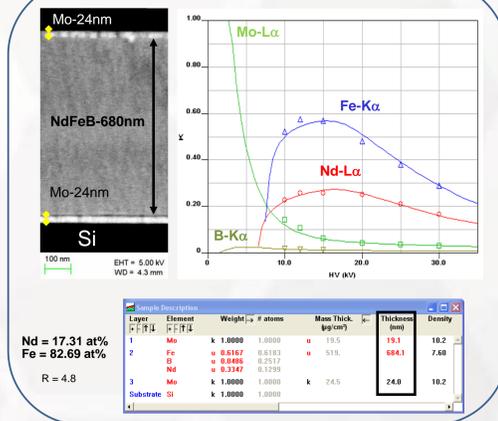
Thin film specimen : Si / Mo / Nd,Fe,B₂ / Mo (4 < y/x < 5); characterisation was carried out on samples in the as-deposited state (i.e., not annealed).



Results on the JEOL840 / EDS Si(Li)



Results on the EPMA JEOL 8800



Estimates of the film thickness and composition (iron relative to neodymium) made with both Si(Li) and EPMA analysis are in good agreement with direct observation / measurements. However, for the boron content, the estimate is greater than expected. Several reasons could explain this. For these runs, no energy filter on the LDE3H spectrometer was used. The Molybdenum layer also creates a convolution between the B-K α and Co-M peaks, and no overlapping correction was applied. Therefore, a weak error occurred but with the M.A.C. and the $\Phi(\rho z)$ correction, the error increases.