MAGNETIC PROPERTIES AND STRUCTURE OF Sm-Fe-N NANO-DROPLETS

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Abstract

Sm-Fe thin films were deposited on a Si wafer coated with ~ 100 nm layer of Ta by ablating different alloys used as solid targets. Further on, based on the results of our previous work, where the coercivities of powders obtained from basic Sm-Fe alloy were significantly improved by additive elements also $Sm_{13.8}Fe_{82.2}Ta_{4.0}$ alloy was used as additional target to prepare thin films and to achieve high coercivities.

Targets were ablated using a molecular fluorine laser at 157 nm at low laser energy of 25 mJ per pulse. Amorphous films or deposited layers of nanocrystals were produced in accordance with the different working conditions and background gasses used. The dimensions of the deposited films or layers of nanocrystals on the Si-Ta substrate varied between 10-500 nm. The composition of the nanocrystals grown by Pulse Laser Deposition (PLD) remains the same as the initial target composition, in contrary to the growth using PLD at longer wavelengths. The magnetic properties (measured by VSM), and the morphology and type of the films (observed and analysed by SEM/EDX), varied significantly with different experimental conditions.

Experimental

- The mechanism that leads to material ablation depends:
  - Laser characteristics
  - Properties of the target
  - Properties of the substrate
  - Distance between the target and the substrate
  - Background gas (type and pressure)

- F_2 Laser: Λ = 157nm, E = 20mJ/pulse, 20 Hz repetition rate
- Target : $Sm_{13.7}Fe_{86.3}$, $Sm_{13.8}Fe_{82.2}Ta_{4.0}$
- Substrate : Si + Ta
- Background gas: HV 10^{-6} mbar, He 1 bar, N_2 1 bar

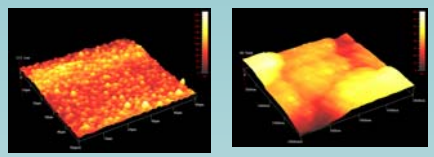
Results and discussion

AFM analysis of substrate surface

Substrate: Si

Sputtered Ta: Sputtering apparatus CemeCon CC800/7 (working pressure 1-2·10^{-3}mbar)

Results:100 nm Ta film

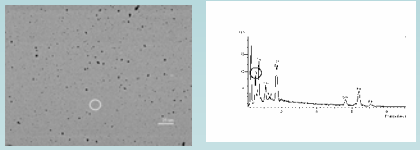


SEM/EDX analysis

Substrate: Si + 100 nm Ta. Target: $Sm_{13.7}Fe_{86.3}$

Background pressure: high vacuum (10^{-6}mbar)

Results:Amorphous film, with droplets of ablated material (X-ray analysis on the film)

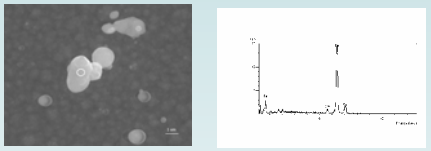


SEM/EDX analysis

Substrate: Si + 100 nm Ta

Background pressure: high vacuum (10^{-6}mbar)

Results: Amorphous film, with droplets of ablated material (X-ray analysis on a droplet)

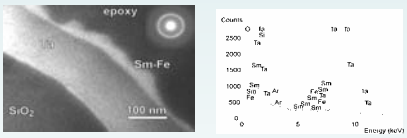


FEG – TEM / (EELS analysis)

Substrate: Si+Ta

Background pressure: high vacuum (10^{-6}mbar)

Results: Amorphous Ta and Sm-Fe layers (selected area electron diffraction pattern)



Experimental + SEM analysis

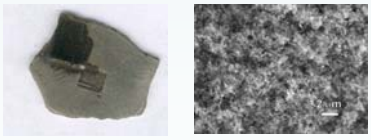
Conditions:

Substrate: Si + Ta. Target: Sm13,7Fe86,3

Background gas: He 1 bar

Results:

Ablated Sm-Fe target (computer-controlled micrometric X-Y-Z translation stage), deposited nanoparticles



Experimental + SEM/EDX analysis

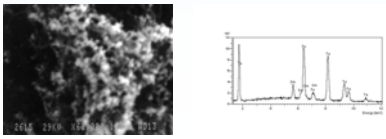
Conditions:

Substrate: Si + Ta. Target: Sm13,7Fe86,3

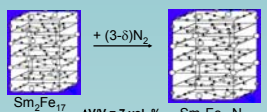
Background gas: He 1 bar

Results:

Deposited nanoparticles have the composition very close to the target composition



Interstitial modification with nitrogen

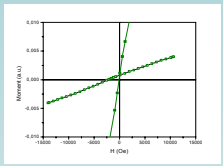


T_c =116°C
PLANAR magnetocristaline anysotropy

T_c=476°C
AXIAL magnetocristaline anysotropy

Magnetic measurements – VSM

Before annealing and nitriding: Hc= 100 Oe



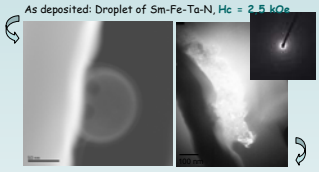
Experimental +TEM examination

Conditions:

Substrate: Si + Ta. Target: Sm13.8 Fe82,2Ta4,0

Background gas: N_2 1 bar

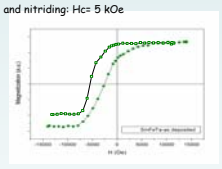
As deposited: Droplet of Sm-Fe-Ta-N, Hc = 2,5 kOe



After annealing and nitriding, nanocrystalline thin layer, Hc = 5 kOe

Magnetic measurements – VSM

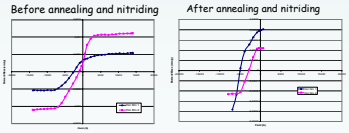
Before annealing and nitriding: Hc= 2,5 kOe After annealing and nitriding: Hc= 5 kOe



Magnetic measurements - VSM

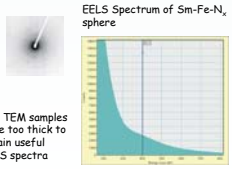
Before annealing and nitriding

After annealing and nitriding



Distance between target and substrate 1.5 cm
Laser energy 40 mJ, growth for 45 minutes (Sample 1)
Distance between target and substrate 0.5 cm
Laser energy 40 mJ, growth for 60 minutes (Sample 2)

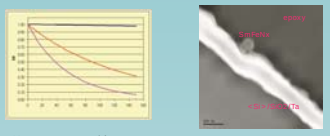
Electron Energy Loss Spectroscopy



The TEM samples were too thick to obtain useful EELS spectra

EDXS analysis – detection of nitrogen

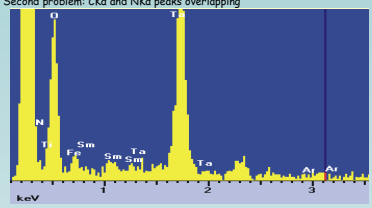
Main problem: Absorption of NKa in C (and O)



Very serious problem in measuring the nitrogen content is the absorption of Nitrogen Ka line in Carbon

EDXS analysis – detection of nitrogen

Second problem: CKa and NKa peaks overlapping



To calculate the absorption:

The thickness of carbon containing layer should be estimated.

In the system LDL for N is around 1,5 wt%, taking into account the absorption in carbon containing layer, the LDL is around 3%

In $Sm_2Fe_{17}N_3$ there is 3,4 wt% of nitrogen

Nitrogen determination

- We can in most cases hardly estimate by EELS the presence of nitrogen in $Sm_2Fe_{17}N_x$ spheres
- With EDXS analysis the complication is that N_{Ka} and C_{Ka} peaks overlap
- Indirect magnetic measurements, using VSM is in this case much more sensitive method for the determination of nitrogen content

Conclusions

- Thin films on the basis of Sm-Fe-(Ta)-N were fabricated by PLD at 157 nm, on Si+Ta substrate in different background pressure and different conditions:
- Atmosphere: HV (10^{-6} mbar): amorphous film (10-30 nm), O_2 contamination
- Atmosphere: He: nanoparticles (50-500 nm) *with a composition close to the composition of the target* (due to the low energy), and with the Hc of 2,5 kOe (after further nitriding)
- Atmosphere: N_2: nanocrystals (~10 nm) formed already during the ablation process with coercivity (Hc) of 2,5 kOe without further treatment confirms that the nitriding starts in the plum.