

Structural and compositional properties of Sm-Fe-Ta magnetic nanospheres prepared by pulsed-laser deposition at 157 nm in N₂

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Sm-Fe-Ta-N magnetic nanodroplets were fabricated by pulse laser deposition at 157 nm at low laser energy of 10 mJ per pulse in nitrogen background pressure. The target intermetallic alloy with the composition of Sm_{13.8}Fe_{82.2}Ta_{4.0} was fabricated by arc-melting and further annealing to achieve homogeneity. The nanodroplets were deposited on a Si substrate coated with a ~ 100 nm thick layer of Ta to avoid chemical reaction between the highly reactive Sm and Si substrate.

The average composition of the deposited material reflected the stoichiometry of the initial target. As prepared droplets were analyzed by the magnetic measurements using SQUID, nano-structures observation was performed by using field emission gun scanning electron microscope (FEG SEM) imaging equipped by electron dispersive X-ray spectroscopy (EDXS), which at the same time allows for the chemical analysis of the composition of the films. The surface morphology of the films was investigated also with AFM and MFM.

For more detailed analyses nano-spheres were examined by a field-emission electron-source high-resolution transmission electron microscope HR-TEM Jeol 2010F equipped with an energy dispersive x-ray spectroscopy (EDXS) (LINK ISIS EDS 300) and electron energy-loss spectroscopy (EELS) (Gatan PEELS 667). The magnetic response on individual nanospheres was detected and quantified for the first time in this system by applying **Electron Holography**.

Nanodroplets with the diameter between 5 nm up to 130 nm were observed with the HRTEM (Figure 1). Small spheres (<50 nm) were mainly amorphous, while larger spheres have a core-shell structure with a crystalline nucleus surrounded by an amorphous layer. (Fig2). The combined EDXS and EELS analysis confirmed that the composition of the inner crystalline part corresponded to the Sm₂(Fe,Ta)₁₇ phase ($\text{Sm}_{(\text{at.}\%)} / (\text{Fe} + \text{Ta})_{(\text{at.}\%)} = 0.11$) enriched with nitrogen. The outer amorphous layer was oxygen-rich with no detectable amounts of nitrogen and significantly different ratio of $\text{Sm}_{(\text{at.}\%)} / (\text{Fe} + \text{Ta})_{(\text{at.}\%)} = 0.64$ (Fig. 3).

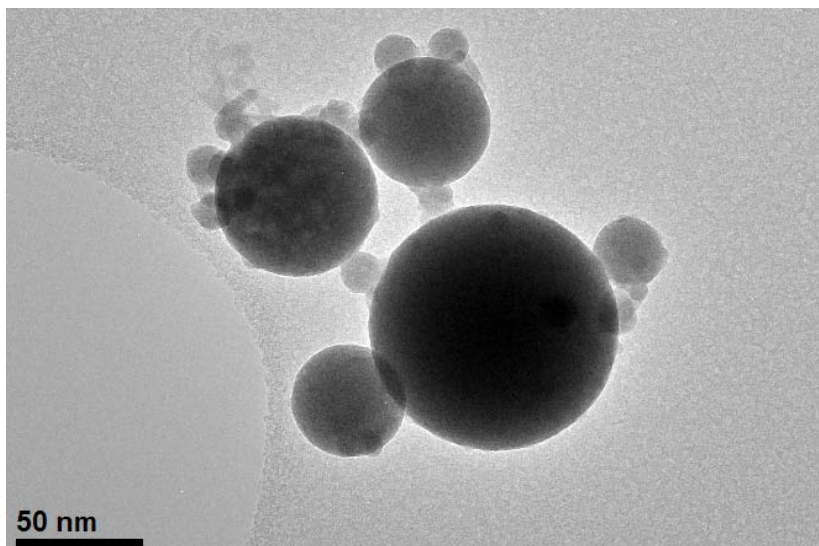


Figure 1. Core-shell structured Sm-Fe-Ta-based nano-spheres in-situ nitrided in Pulsed Laser Deposition at 157 nm.

These analytical results prove that nitriding of nanodroplets was taken place already in the plume and thus further post ablation annealing of the films was unnecessary for ferromagnetic response. The liquid nanodroplets solidify on the surface of a Si-Ta substrate in two phases. One is a crystalline nucleus, which is surrounded by an amorphous one. The bi-phase spherical nanostructure retains its ferromagnetic response of its crystal nucleus far more efficiently than the high porosity crystal structures grown at higher laser energies because of their accelerated oxidization.

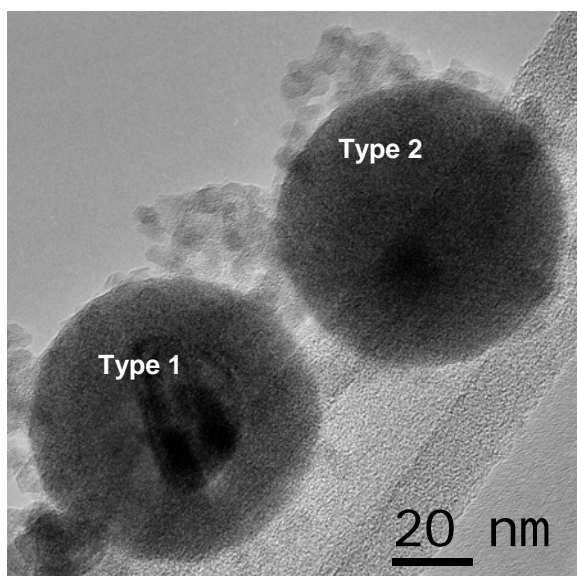


Fig. 2. Nanospheres obtained by in-situ nitrogenation in PLD processing

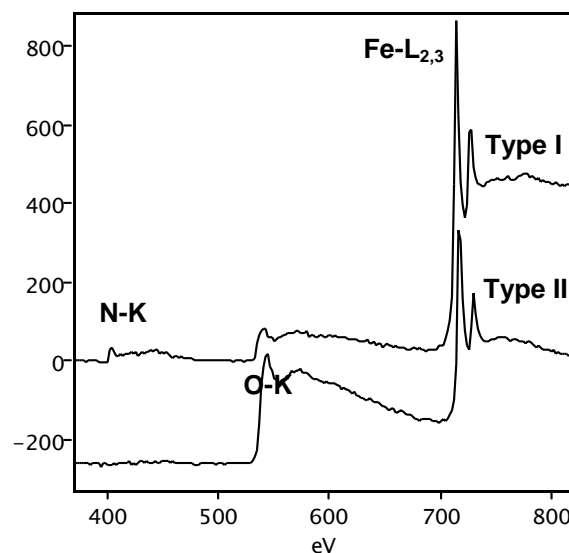


Fig. 3. EELS spectra of type I and Type II nanospheres