(S)TEM/EELS characterization of phase and structural state in $Fe_{88}Zr_{10}N_{11}$ ferromagnetic films

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A new generation of magnetic films with required complex of properties determines in great extent a success in the development of the modern microelectronics. In this connection an attention of the researches is focused on investigations of nanocrystalline magnetic soft films based on Fe-Me-X system (X=B, C, N, O и Me=Ti, Zr, Hf, Nb, Ta) with unique combinations of properties, such as high saturation induction (up to 2T), low coercive field (<0.1 Oe), high thermal stability. [1].

In this work we report about a structural characterization of $Fe_{88}Zr_{10}N_{11}$ ferromagnetic films fabricated by a reactive rf magnetron sputtering technique on heat resistant glass substrates and annealed at temperatures in the range of T=200-650^0 C at residual pressure of 3×10^{-6}Torr. The films have a thickness of about 1-2 μm.

Cross sections for (S)TEM and HREM investigations were prepared by ion milling in Gatan PIPs 691. All samples were characterized in a Tecnai G^2 30ST at accelerating voltage of 300kV. EELS-mapping was carried out in a Tecnai F20 XTwin equipped with a Gatan Tridium 863 GIF and high-angle-annular dark-field (HAADF) STEM detector. The probe size used for the experiment was 1 nm. The images and SAED patterns were processed and analyzed with the Gatan Digital Micrograph 3.11.1 software and JEMS package [2].

Nitrogen-oversaturated α –Fe phase with sizes in the range of 2-5 nm was observed in the structure of as-sputtered films, fig.1. ZrN-crystals appeared after annealing at the T=200^{0}C. Such kind of structural state remained stable till temperature of annealing of T=600^{0}C. At high temperatures of heat treatment we observed nanocrystals of α –Fe, γ –Fe (for some films), ZrN and perhaps both Fe_2N and Zr_1O_{x-1} in accordance with SAED patterns, DF TEM and HREM images, fig.2. α –Fe and ZrN phases exhibited a texture. Nanocrystalline conglomerates formed column structures (white arrows, fig.2) grown out of substrate.

EELS-mapping of the film annealed at T=600^{0}C demonstrated compositional heterogeneity in the film-substrate interface, fig.3: Zr-atoms diffused inside of substrate, while Fe-atoms gathered on the interface regions. The film was impoverished by nitrogen in comparison with the substrate in the region of interface. Correlation of the compositional heterogeneity both zirconium and oxygen was observed.

In this connection we can suggest that Fe_2N-phase formed as a result of decreasing of the N-content in the α –Fe crystals at high temperature of annealing; Zr_1O_{1-x} phase formed as a result of interaction with substrate that could become the reason of occurrence of γ –Fe phase with the same interplanar spacings.

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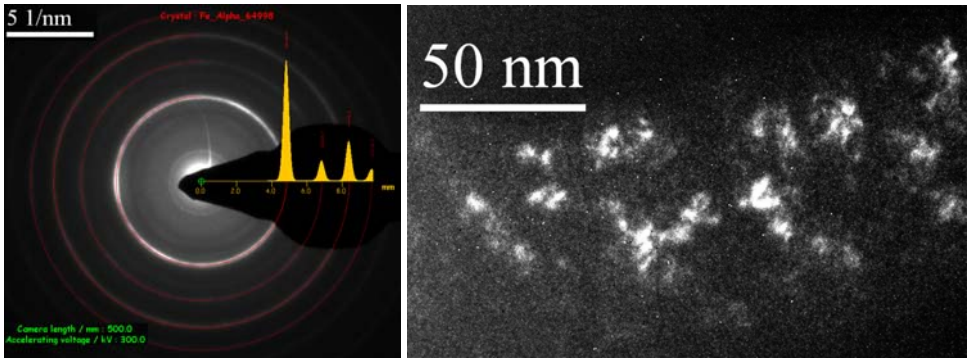

Figure 1. Structure of as-sputtered film: DF TEM image and the corresponding electron diffraction pattern (SAED) with calculated pattern.

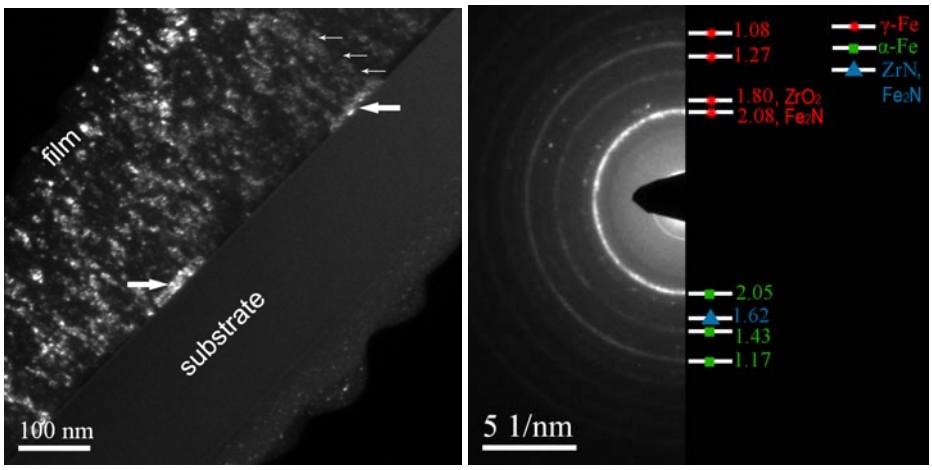

Figure 2. Structure of the film annealed at T=650^{0}C: DF TEM image and the corresponding electron diffraction pattern (SAED).

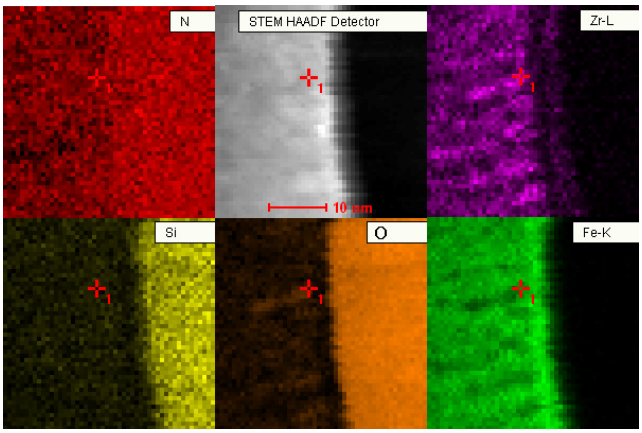

Figure 3. EELS-mapping for the film annealed at T=600^{0}C.

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