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In-Situ Growth of MnAs nanocrystals in Si studied by Transmission Electron Microscopy

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The growth of magnetic nanocrystals inside a semi-conducting matrix is aimed to the development of new devices for data information storage and high sensitive magnetic sensors [1]. As properties of nanostructured materials may differ from their bulk materials in terms of magnetic anisotropy, magneto-resistance, Curie-temperatures and susceptibility [2, 3], careful characterization on the nanometer scale is required. Theoretical studies propose ferromagnetism for transitional metal-doped Si [3] and SiC [4] with impurity concentrations above 3%. We showed the formation of magnetic nanocrystals after high dose (upto $10^{17}cm^{-2}$), high temperature metal ion implantation into SiC followed by rapid thermal annealing [5].
In this work we study in-situ formation and growth of Mn/As nanocrystals within crystalline Si. Mn and As ions have been co-implanted at 350°C into Si wafers.
Proper TEM sample preparation is a crucial task because the process has to be studied in cross-sectional view and the sample must withstand temperatures up to 1000°C. Cross-section TEM samples have been prepared using the following methods and techniques: A ceramic based glue (for high temperature treatment) has been used to prepare the sandwiches. The thin TEM sample was made by using mechanical grinding, dimpling and polishing, followed by low angle Ar-ion etching. Plasma cleaning is necessary to remove any residual carbon hydrogens from the sample surface to avoid contamination during heating. TEM investigations were carried out using a Philips CM20 microscope together with a Gatan heating stage sample holder (specified to 1000°C). Additional investigations (tomography, HRTEM, EFTEM) were carried out using a image side C_S-corrected FEI Titan.
Fig. 1a shows the Si sample for the as-implanted case. The Si-substrate contains defects up to a depth of 200nm caused by ion implantation such as stacking faults and dislocations. Si itself is crystalline due to high temperature ion implantation. No nanocrystals can be found in the as-implanted sample. Fig 1b shows the same sample position after heating the specimen up to 950°C in small steps of 15°C/min. As it can be seen nanocrystals have been formed. HRTEM investigations show that the nanocrystals have a separate phase and are facetted. In addition, the three-dimensional shape of the nanocrystals could be revealed by electron tomography. Fig. 2 shows visualizations of a reconstructed tomogram of one of the formed MnAs nanoparticles. The in-situ observations on the sub-nano scale revealed directly for the first time that the precipitates form at the defect [6].

[1] Pokrant S., Herwig C., Hihara T., and Becker J. A., *Eur. Phys. Journ.* **D 9** (1996), 509
[2] Leslie-Pelecky D. L., and Rieke R. D., *Chemistry of Materials* **8** (1996), 177

[3] Shi J., Gider S., Babcock K., Awschalom D. D., *Metals and Semiconductors Sc.* **271** (1996), 937
[4] Shaposhnikov V.L., Sobolev N.A., *Journal of Physics Condensed Matter* **16** (2004) 1761
[5] Biskupek J., Kaiser U., Lichte H., Lenk A., Pasold G., Witthuhn W., *Magnetism and Magnetic Materials* **293** (2005) 394
[6] Kaiser U., Muller D.A., Grazul J.L., Chuvilin A., Kawaskaki M., *Nat. Mat.* **1-2,** (2002) 102-105
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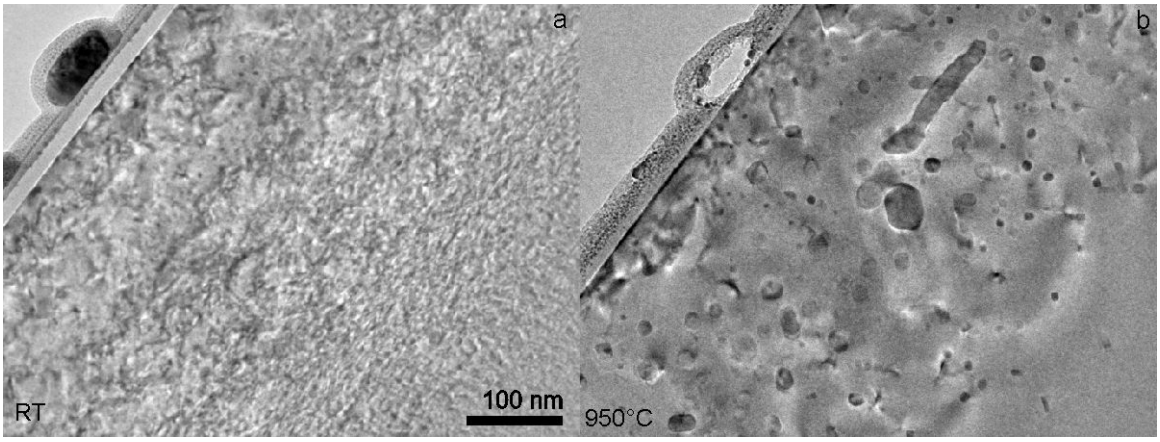

Fig.1. (a) Mn/As-implanted Si at room temperature. Only implantation defects are visible in the sample (At the surface are some residuals of the ceramic glue left)
(b) The sample after in-situ annealing at 950°C. Nanocrystals have been formed (dark contrast due to absorption and bragg-contrast). The implantation defects in the Si-substrate are annealed.

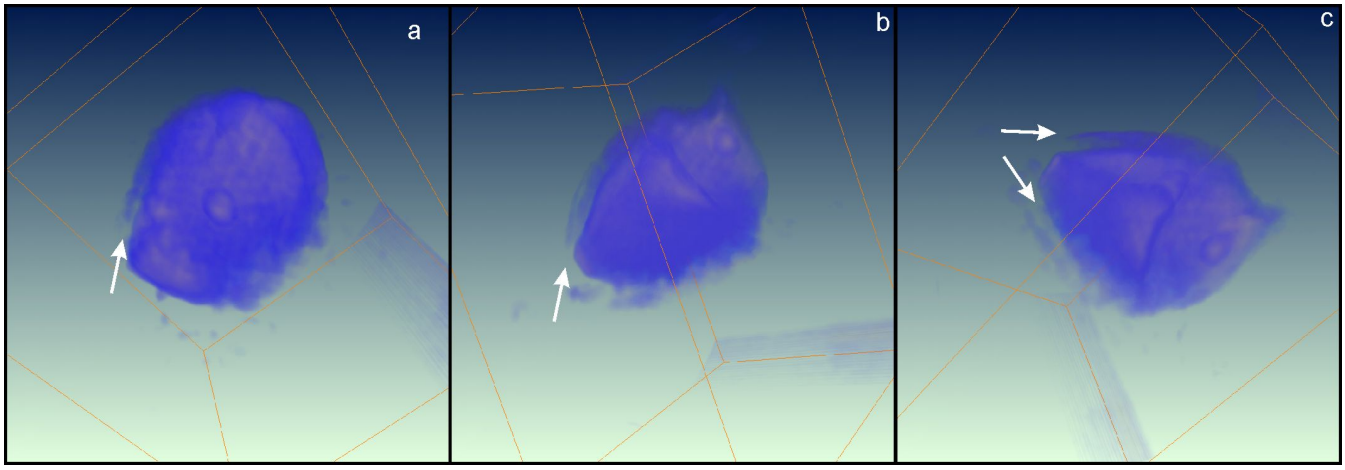

Fig.2. 3D-visualization of an in-situ grown MnAs nanoparticle. HRTEM, EFTEM and EELS analysis show that the particle contains a crystalline Mn-rich core and a shell of an amorphous As-rich phase. The arrows indicate the amorphous As shell.