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3D Imaging of Si and Er Nanoclusters in Er Doped $SiO_{1.5}$ Films by STEM Tomography

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The photonic properties of nanoscale silicon particles suggest numerous optoelectronics applications [1,2]. Silicon nanoclusters (NCs) also act as powerful sensitizers for the luminescent transitions of the rare earth ions [3]. Various TEM techniques such as HRTEM and EFTEM [4,5] have been used to study fundamental microstructural properties of silicon nanocomposites. Here we introduce a STEM technique that is suitable to study Si NCs with heavy element dopants, especially combined with TEM tomography to investigate the cluster size, shape and spatial distribution of both Si and dopant nanoclusters in three dimensions.

We have investigated thin erbium-doped silicon-rich oxide (SRO) films. The samples were deposited onto Si substrates by simultaneous thermal evaporation of silicon monoxide (SiO), silica (SiO_2), and metallic erbium (Er). In situ crystal monitor readings estimated a film composition of $SiO_{1.5}$ (10 at.% excess Si) with an erbium doping level of about 6 x 10^{20} cm^{-3} (1 at.%), and a film thickness of 200 nm. Subsequent annealing in forming gas at 1000°C causes the Si and SiO_2 phases to segregate and induces growth of silicon nanocrystals, but also promotes erbium clustering. TEM cross section samples were prepared by mechanical polishing and by Ar ion milling with an incident energy of 1.0keV and incident angle of 8 degrees. Needle-shape tomography samples were sectioned by 30keV Ga ions on a Zeiss Nvision40 focused ion beam (FIB) system, and then mounted onto an Omini TEM grid by the in-situ lift-out technique. A pillar with a diameter less than 100nm was polished with low current probes (<1pA). Ar ion milling with low incident angle and low energy (6 degrees and 0.5keV) at the final stage was used to reduce the amorphous layers introduced in the FIB process [6].

A JEOL 2200 FS TEM equipped with an Omega filter lens was used to image both Er and Si nanoclusters in $SiO_{1.5}$:Er films. Fig. 1. shows the positions of STEM detectors in a JEOL 2200 FS TEM. Energy filtering both the BF and DF STEM images, together with simultaneous acquisition of HAADF, allowed us to determine relative location of Er and Si NCs. We have used the silicon plasmon energy [4] for energy filtered STEM images. High angle annular dark field (HAADF) (above the energy filter) STEM images provide atomic number contrast for Er clusters. Therefore two elements (Si and Er) can be detected simultaneously within one scan with two detectors in STEM images (Energy Filtered DF/BF and HAADF) with no image shift between tthem. Fig. 2. shows a superposed image using both HAADF and DF detectors (energy filtering at the Si plasmon energy) showing Er and Si contrasts respectively. However the sizes and positions of Er and Si clusters in the 2D projection image can be easily misinterpreted due to overlap.

TEM tomography was used to investigate the relative location of Si and Er nanoclusters in three dimensions. An image-tilt series was recorded and reconstructed with TEMography™ software. A custom designed sample holder with ±90° tilt allowed us to collect data without a missing wedge. A

FIB-prepared TEM sample with needle shape (shown in Fig. 3.) provides no thickness increase with increasing tilt angle, eliminating the limitations typical for a thin film sample. Fig. 4. shows a superposed image of Si and Er nanoclusters from a FIB-prepared needle-shape sample. Image series of HAADF and Energy Filtered DF STEM images were reconstructed separately and combined after reconstruction.

References

[1] J. Carreras, J.Arbiol, B. Garrido, C. Bonafos, J. Montserrat, Appl. Phys. Lett., 92, 091103-1-3 (2008).
[2] H. Lee, J.H. Shin, N. Park, Optics Express 13, 9881 (2005).
[3] A. Meldrum, A. Hryciw, A.N. MacDonald, C. Blois, T. Clement, R. DeCorby, J. Wang, Q. Li, J. Lumin 121, 199 (2006).
[4] A. Yurtsever, M. Weyland, and D. Muller, Appl. Phys. Lett. 89**,** 151920 (2006).
[5] S. Schamm, C. Bonafos, H.Coffin, N. Cherkashin, M.Carrada, G. Assayag, A. Claverie, M. Tence, C. Colliex, Ultramicroscopy, 108, 346-357 (2008).
[6] A. Barna, B. PCcz, M. Menyhard, Ultramicroscopy, 70, 161-171 (1998).

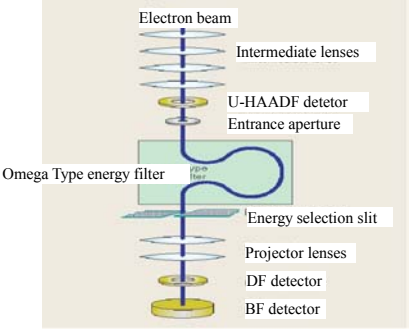

Fig. 1. Positions of STEM detectors in a JEOL 2200FS TEM.

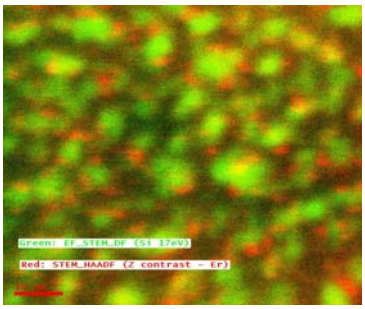

Fig. 2. A color mixed image showing sizes and positions of Er and Si NCs in 2D STEM images taken from a cross section TEM sample *(green is Si; red is Er, scale bar = 10nm)*.

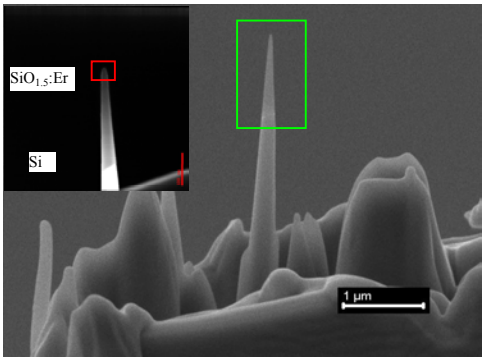

Fig. 3. An SEM image of a needle-shape TEM sample for tomography prepared by FIB. Insert is an EFTEM image at the Si plasmon energy of the area in the green box. Area imaged in STEM tomography is in the small red box *(scale bar = 500nm)*.

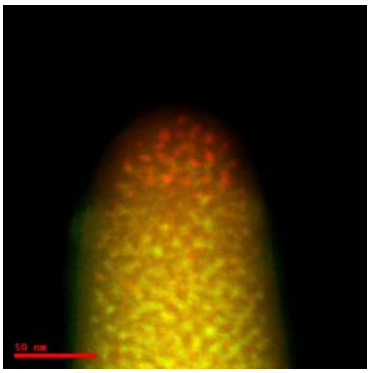

Fig. 4. A color mixed image taken from a FIB-prepared needle-shape sample (*green is Si; red is Er, scale bar = 50nm)*.

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