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Electron Tomography of Si and Er Particles in SiO_x Film Without Missing Wedge.

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Electron tomography (ET) in a TEM has been widely used to characterize materials at sub-10 nm spatial resolution. As any technique, ET has its limitations. One of the most serious problems is the "missing wedge" in Fourier space that arises from limited tilt angle range in a TEM. The most direct way to solve the problem is to tilt of sample over the full tilt range (-90° to 90°). The full tilt range ET was reported for example by Jinnai et. al. [1,2], who analyzed polymer samples with metal nanoparticles. Here we report full tilt chemically selective ET of sub-10 nm Er clusters and Si crystallites embedded in SiO_x matrix. The studied material is a possible candidate for fabrication of Si-based opto-electronic devices [3].

The samples were prepared by deposition of silicon monoxide (SiO), silica (SiO_2), and metallic erbium (Er) onto Si substrates by simultaneous thermal evaporation. 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} (5 at.%), and a film thickness about 200 nm. Subsequent annealing in forming gas at 1000°C caused the Si and SiO_2 phases to segregate and induced growth of silicon nanocrystals, but also promotes undesirable erbium clustering.

A 100 nm diameter rod sample, prepared in a Hitachi NB 5000 FIB/SEM, was welded onto a Hitachi 3D analysis holder that can be inserted into both TEM and FIB [4, 5]. The sample was then analyzed in a Hitachi HF 3300 TEM without demounting from the holder. Figure 1 shows SEM images of the rod shape sample prepared in FIB. High angle annular dark field (HAADF) STEM images were used to image Er clusters at 300 kV, as shown in Figure 2. A tilt series of a full turn (0° to 180°) were taken at 1° increment. ET reconstruction was performed using TEMographyTM package [6]. The major challenge in reconstruction of this sample stems from the overlap of multiple Si crystallites in each projected image leading to difficulties in image alignment. Since we are interested in shape of *individual*, sub-10 nm Si crystallites and Er clusters and their mutual position in 3D the requirements on the quality of the reconstructed ET are extremely strict. The need to retrieve the 3D shape/sizes and relative positions of Si NPs and Er clusters are necessary to quantitatively explain the light emitting properties of this material. Possible way to reconstruct Si is to image and construct Si with energy filtering imaging at Silicon plasmon energy window [7]. Since post-acquisition alignment of Er and Si signals recorded separately is always subject to uncertainties, it is preferable to acquire Er and Si signals *simultaneously* using high-angle annular dark field and energy filtering bright field STEM for Er and Si signals respectively [8].

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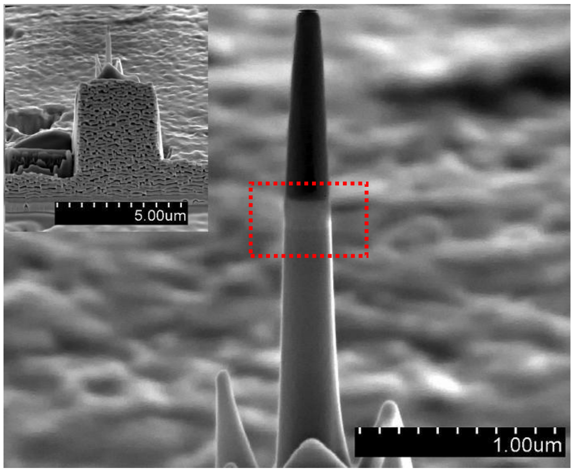

Figure 1. SEM image of a rod shape sample prepared in FIB. Insert shows a low magnification Image. (Er-doped Si layer is marked by the red box).

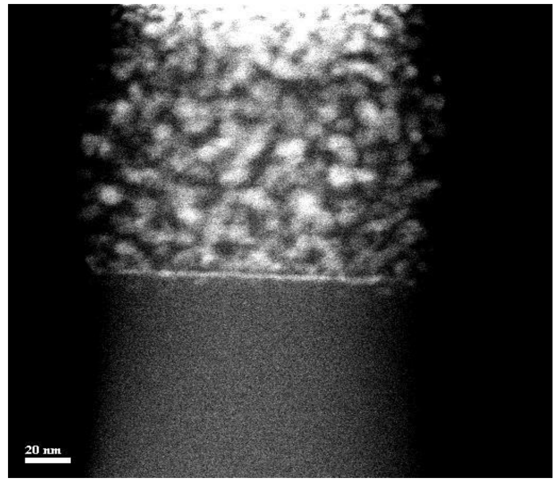

Figure 2. HAADF STEM images of Er clusters in SiO_x matrix.

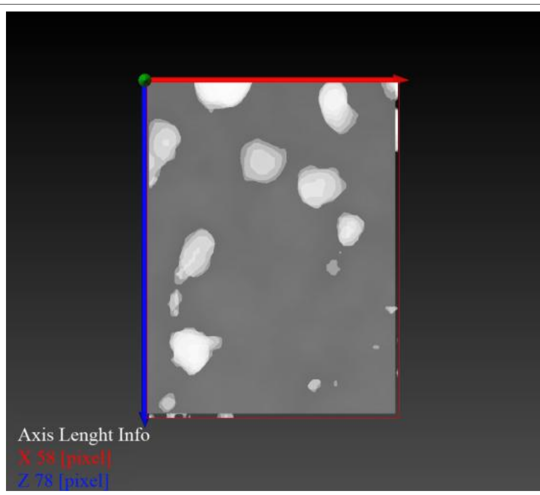

Figure 3. Cross section of the X-Z plane of reconstructed tomogram. The shape is not elongated.

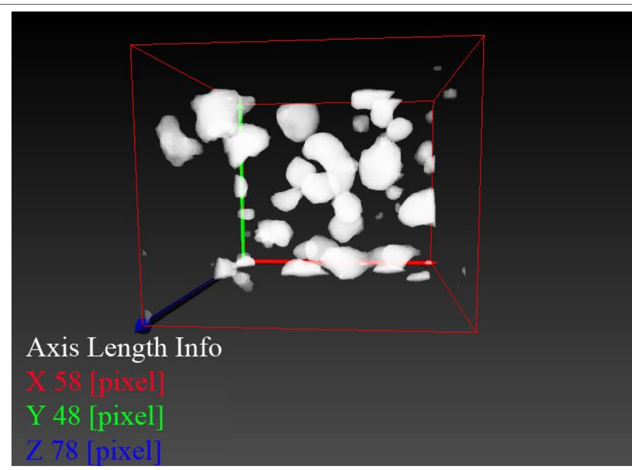

Figure 4. Reconstructed Er clusters

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