Comparative HRTEM studies of radiation effects on FZ-Si samples irradiated with low and high energy electrons.

High resolution transmission electron microscopy investigations (HRTEM) have been performed with the analytical electron microscope JEOL ARM 200F with a resolution of 0.19 nm in the HRTEM mode. The observations were carried out at 200 kV and low current densities, to reduce radiation effects produced by the electron beam. Samples for HRTEM were prepared in cross section, i.e. cutting the Si wafers in (2 x 1) mm slices, gluing them face to face, mechanical thinning to a thickness of ~20 μm followed by ion milling to thicknesses <100 nm, at which they become transparent to the electron beam. The used glue polymerize at room temperature in 3-4 days. In this way we avoid heating the specimens during their preparation for HRTEM.

We have studied the following samples:
1. FZ Si with diffused ^{17}O, irradiated with electrons at a low energy of 3.5 MeV and a high fluence of $1x10^{17}$ cm^{-2};
2. STFZ Si (standard), irradiated with electrons at an energy of 15 MeV and a fluence of $1x10^{16}$ cm^{-2} ;
3. DOFZ Si (with diffused ^{16}O), irradiated with electrons at an energy of 27 MeV and a fluence of $2x10^{16}$ cm^{-2} .

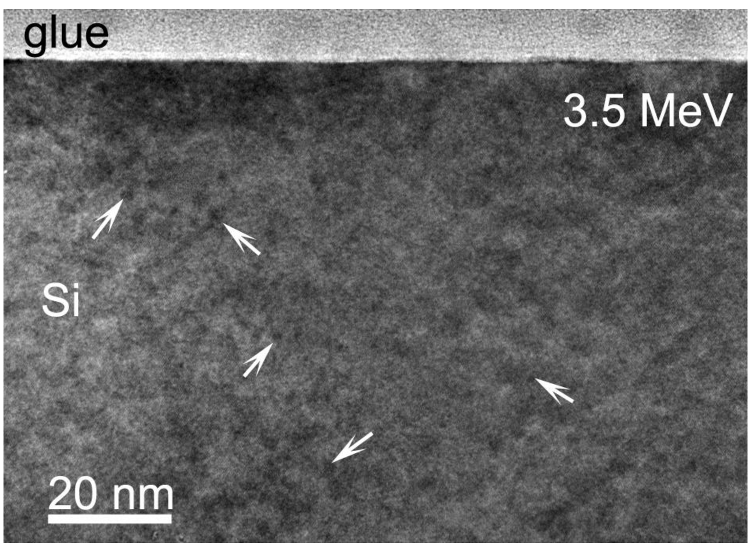

Figure 1. Cross section TEM image on the sample irradiated at low energy (3.5 MeV)

Figure 1 shows the characteristic TEM image of a sample irradiated at a low energy of 3.5 MeV, but a high fluence of $1x10^{17}$ cm^{-2}. One observes the presence of clusters of point defects, vacancies and/or interstitials, visible as dark dots. Some of them are marked by white arrows. Most of them have dimensions <2.5 nm and the largest ones do not surpass 4 nm.

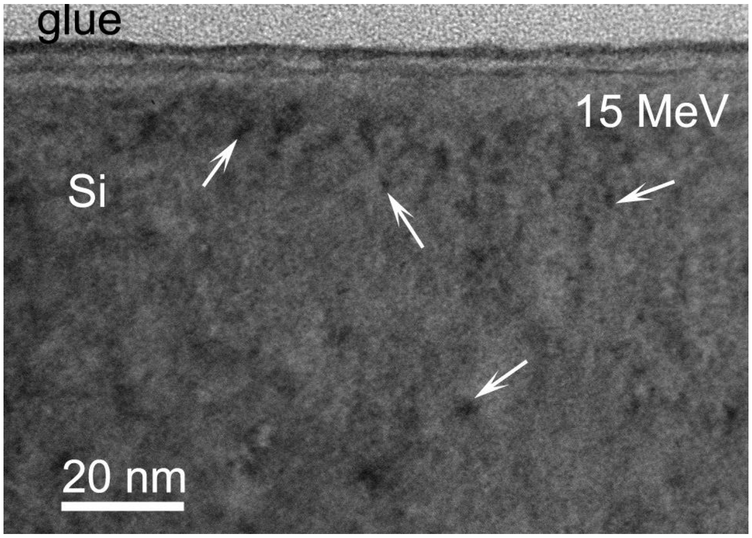

Figure 2.Cross section TEM image of the sample irradiated at 15 MeV.

Figure 2 shows a TEM image of a sample irradiated at a relatively high energy of 15 MeV, but a fluence of one order of magnitude smaller ($1x10^{16}$ cm^{-2}) than the sample irradiated with 3.5 MeV electrons. As in the case of low energy irradiations one can observe the presence of clusters of point defects (some shown with white arrows), having dimensions between 1 and 5 nm, but unless the sample irradiated with 3.5 MeV, in this case, the clusters of larger dimensions (3-4 nm) prevails.

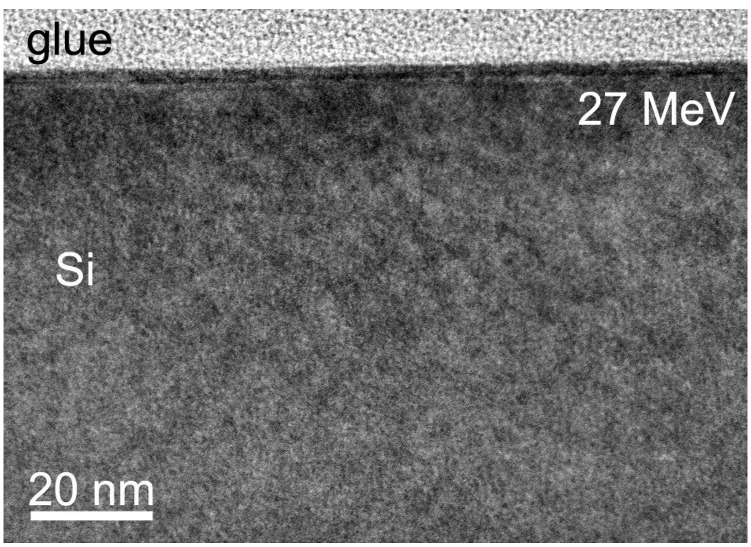

Figure 3. Cross section TEM image of the sample irradiated at 27 MeV.

Figure 3 represents a TEM image characteristic for the sample irradiated with electrons at even higher energy of 27 MeV and a fluence of $2x10^{16}$ cm^{-2}, the double of the fluence of the sample irradiate with 15 MeV. Compared to this sample, the density of defect clusters (black dots) is higher, but their dimension is smaller, approximately 1-2 nm. Also the clusters of points defects are very uniformly distributed and the agglomerates of larger dimensions, as those indicated in figure 2, appear less frequently.

More details of the structure of defects produced by electron irradiation can be obtained by high resolution electron microscopy (HRTEM).

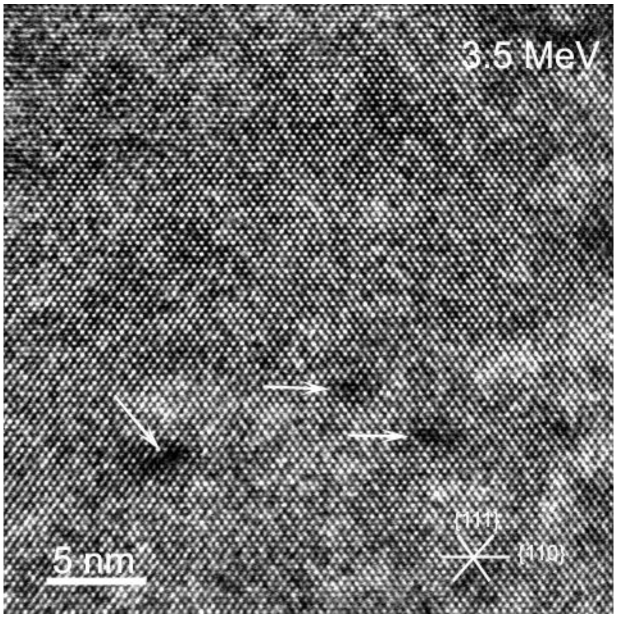

Figure 4. HRTEM image along the [110] zone axis for the sample irradiated with electrons at low energies (3.5 MeV) and a high fluence ($1x10^{17}$ cm^{-2}).

Figure 4 is a HRTEM image along the [110] zone axis of the FZ Si sample with diffused ^{17}O, irradiated with electrons at a low energy (3.5 MeV) and a large fluence ($1x10^{17}$ cm^{-2}), which reveals as dark contrast the presence of the clusters of point defects. For a better understanding of the HRTEM image, some remarks have to be made. The HRTEM image we refer at represent a projection of the Si structure along the [110] zone axis. In the conditions of Scherzer defocus in which we worked, the atoms columns appear as black dots, while white dots represent channels in the structure of silicon. Because point defects appear along the columns of atoms, their clustering is evidenced by dark contrast. From figure 4 it results that, after irradiation appear either small clusters of point defects or single clusters evidenced a single dark dot, or agglomerates of point defect clusters. If the clusters of point defects agglomerate along the principal crystallographic directions, especial the <111> and the <110>, star-like defects or black patches result. Some of the black patches are indicated by white arrows in figure 4. They have dimensions around 3-5 nm. The clusters of point defects are formed by accumulation of vacancies and/or interstitials resulting from the collision cascade. It is interesting to mention that they appeared even in the case of low energy electron irradiations, but at a relatively high fluence.

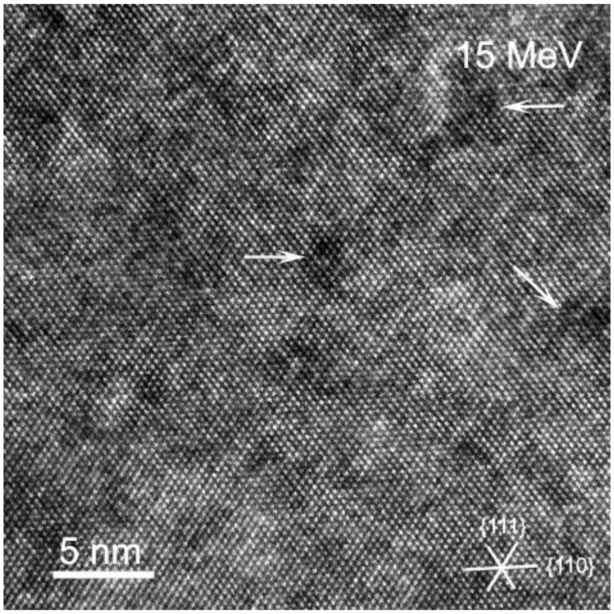

Figure 5. HRTEM image along the [110] zone axis on the sample irradiated with 15 MeV and a fluence of $1x10^{16}$ cm^{-2}.

A HRTEM image of the sample irradiated with electrons at an energy of 15 MeV and a fluence of $1x10^{16}$ cm^{-2} is given in figure 5. As for the sample irradiated at low energy, clusters of point defects appear. Unless this sample, there is a small density of single clusters, most of the agglomerates forming star-like defects and black patches (indicated by with arrows) of larger dimensions (3-6 nm).

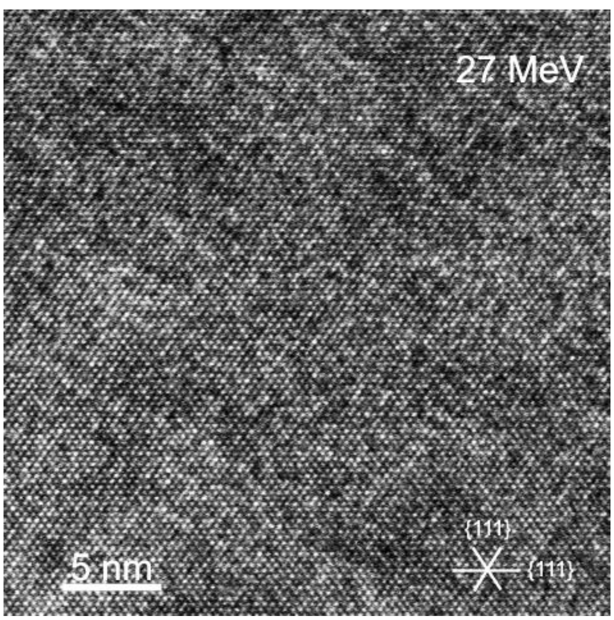

Figure 6. HRTEM image along the [110] zone axis on the sample irradiated with 27 MeV and a fluence of $2x10^{16}$ cm^{-2}.

Figure 6 shows a HRTEM image on the sample irradiated with electrons at high energy (27 MeV) and a fluence of $2x10^{16}$ cm^{-2}. Compared with the two preceding cases, one observes the appearance of agglomerates of small clusters of point defects, relatively uniformly distributed.

Conclusions:
From the TEM/HRTEM studies on the three FZ Si samples irradiated with electrons in different conditions one can conclude the followings:

1. Irradiation with low energy (3.5 MeV) electrons and high fluencies ($1x10^{17}$ cm^{-2}) produces extended defects as clusters of point defects.

2. The irradiation effects of low energy electrons and high fluencies are alike to those produced by electrons accelerated at relatively high energies (15 MeV) and a fluence one order of magnitude smaller.

3. High energy irradiations (27 MeV) and medium fluencies ($2x10^{16}$ cm^{-2}) produce a very high density of agglomerates of point defect clusters.