# High resolution transmission electron microscopy (HRTEM) investigations of silicon irradiated with high energy electrons

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## Studied samples:

1. STFZ Si irradiated at RT with electrons: energy 15 MeV, dose $1x10^{16}$ $cm^{-2}$, subjected to an annealing treatment in the following protocol: RT for ~3months, 80 $^{o}$C for 73380 min. (~51 days) + 200 $^{o}$C for 30 min. + 275 $^{o}$C for 30 min.

2. DOFZ Si irradiated at RT with electrons: energy 27 MeV, dose $2x10^{16}$ $cm^{-2}$, subjected to an isochronal annealing treatment in the following protocol:
80 $^{o}$C for 30 min. + 140 $^{o}$C for 30 min. + 200 $^{o}$C for 30 min.

## Instrument:

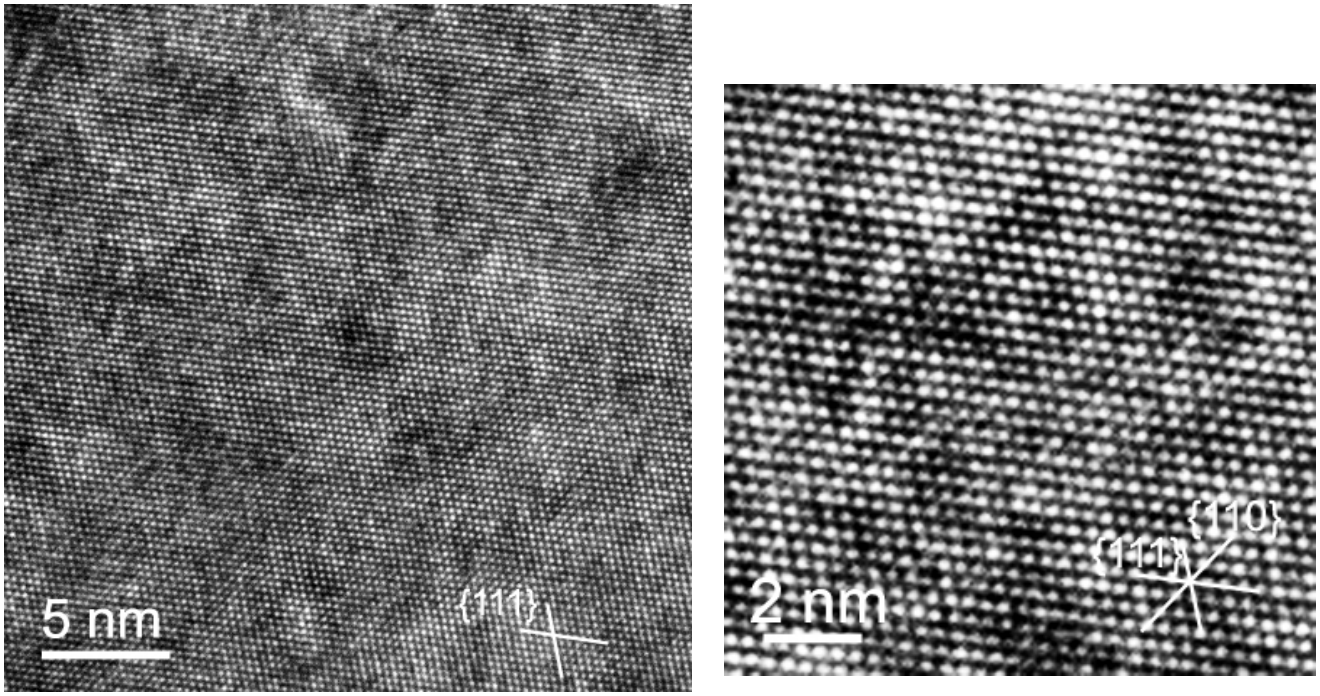
High resolution analytical JEOL ARM 200F operating at 200 kV, with a resolution in the HRTEM mode of 0.19 nm.
HRTEM imaging was performed with the Si specimen oriented along the [110] zone axis.

## Specimen preparation for HRTEM:

- sawing strips from the Si irradiated wafer;
- gluing the strips face to face with a glue which cured at room temperature;
- mechanical thinning followed by ion milling.

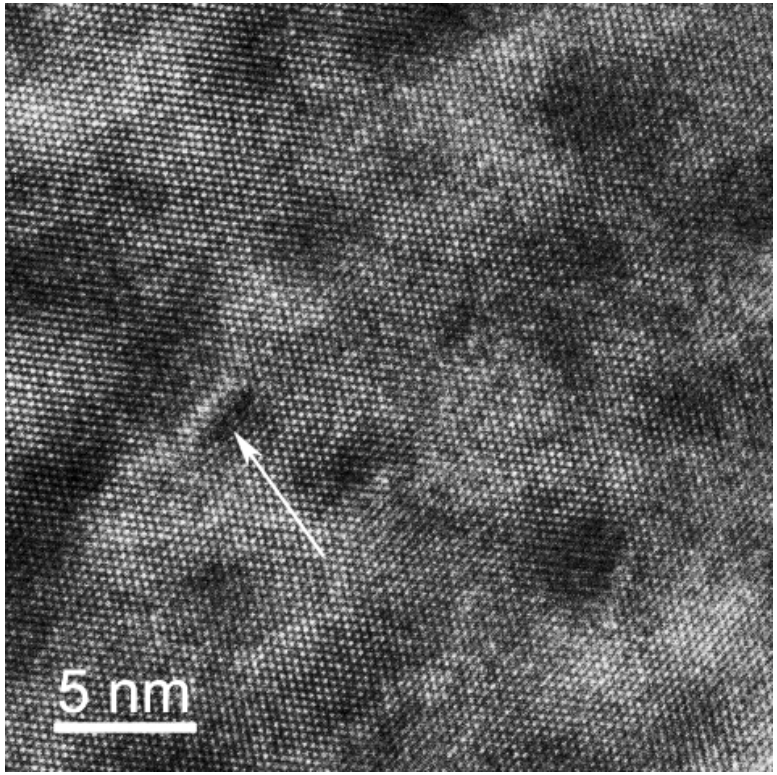
All thermal treatments of the irradiated samples have been performed on the thinned specimens, in a furnace under flowing Ar.

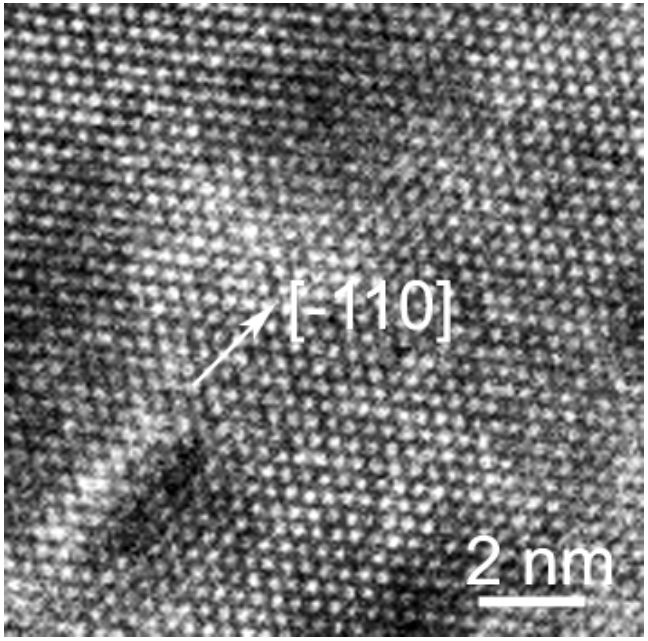
# The 15 MeV irradiated specimen




*HRTEM images of the specimen after electron irradiation*. Clusters of point defects (vacancies and interstitials), some formed along the <111> and <110> directions, are revealed by their darker contrast. Their size is smaller than 3 nm.

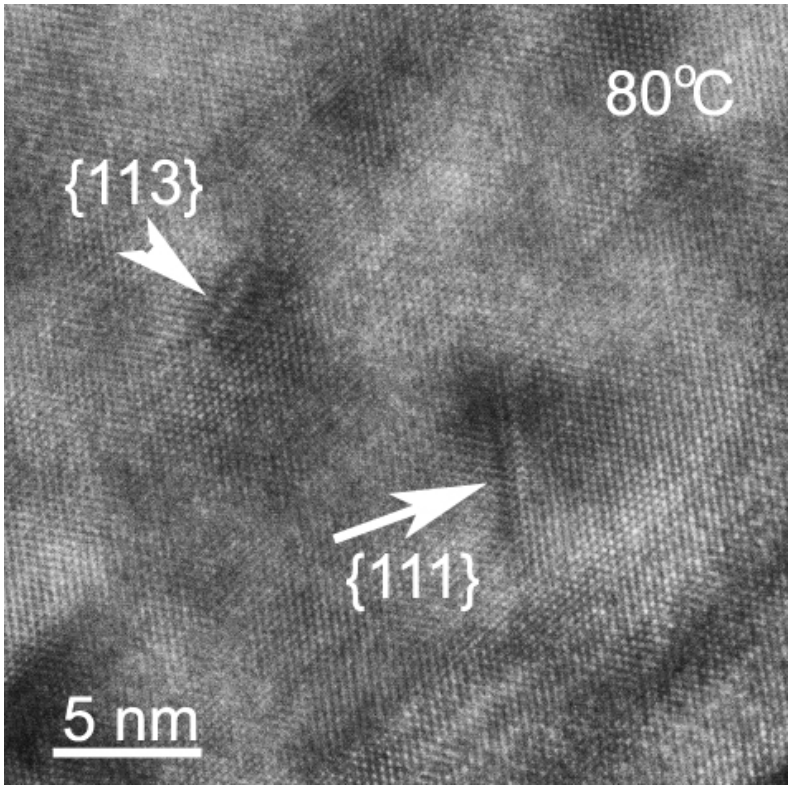
# The 15 MeV irradiated specimen



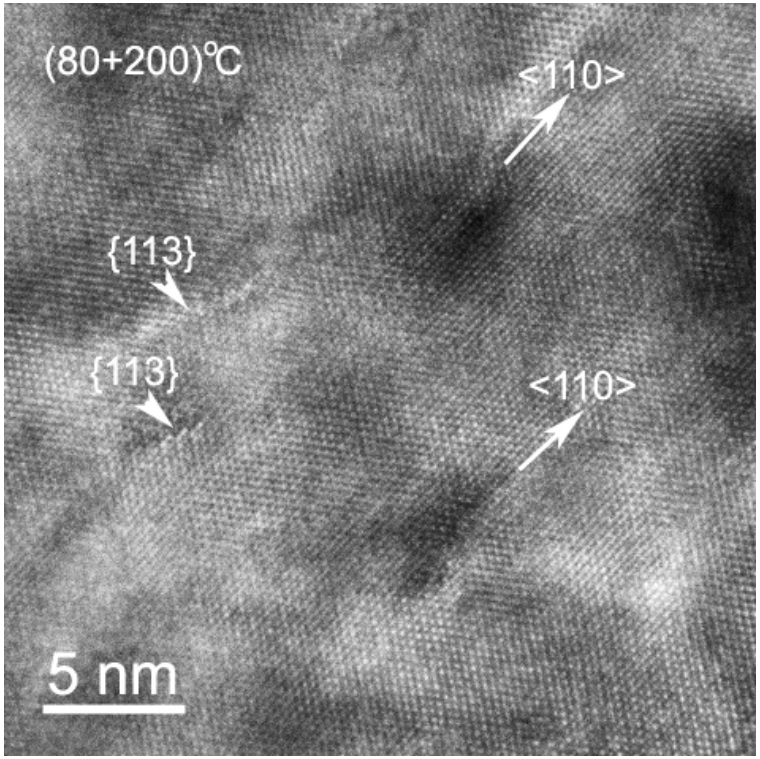

*HRTEM image of the irradiated specimen kept for ~3 months in the TEM holder at RT.* A plate-like defect cluster marked by arrow oriented along the [-110] direction is revealed. Such a defect occurs from the agglomeration of self interstitials [S. Takeda, T. Kamino, PRB 51, 2148 (1995)]. The clusters of point defects are also visible by the dark contrast.

# The 15 MeV irradiated specimen annealed at 80 °C for ~51 days



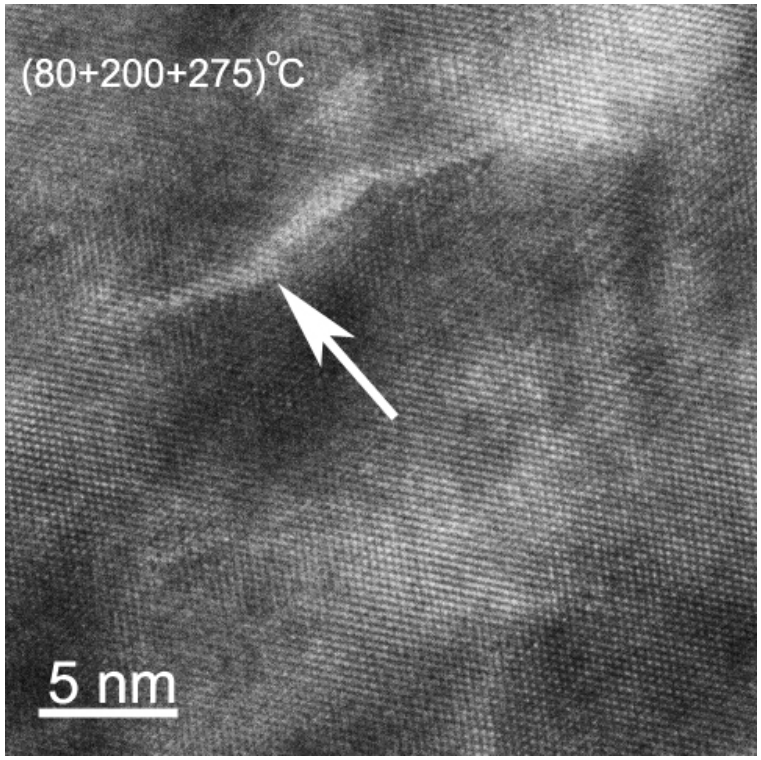

*Long time annealing at low temperatures* determines the formation of extended defects such as the {111} Frank dislocation loop formed by the aggregation of vacancies [L. Fedina et al., Phys. Stat. Sol. (a) 171, 147 (1999)] or the {113} rod-like defect formed by agglomeration of interstitials [S. Takeda, Micr. Res. & Techn. 40, 313 (1998)].

The 15 MeV irradiated specimen annealed at 80 $^{o}$C for ~51 days + 200 $^{o}$C, 30 min.



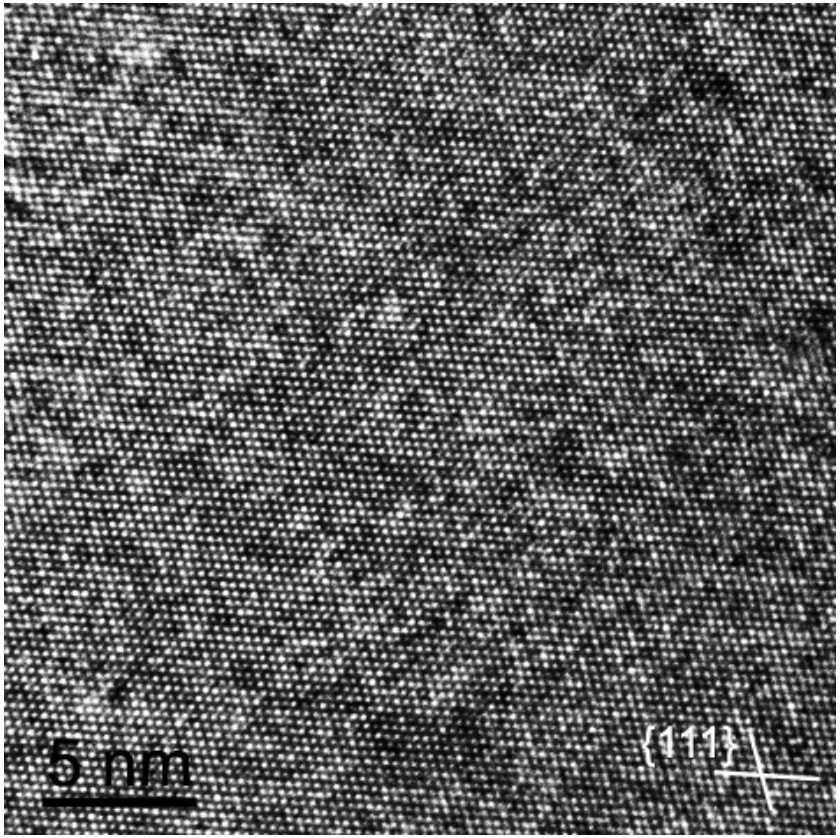

*Further annealing at 200 $^{o}$C* determines the increase in number of the {113}-type rod-like defects. Also defects growing along the <110> directions are observed. These defects identified as agglomerates of self interstitials are the precursors for the formation of the {113} defect [S.Takeda, T. Kamino PRB 51, 2148 (1995)].

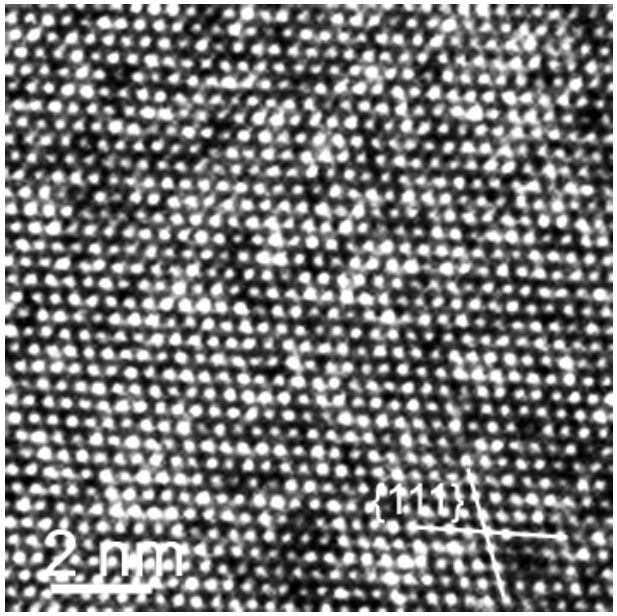
The 15 MeV irradiated specimen annealed at 80 $^{o}$C for ~51 days + 200 $^{o}$C, 30 min. + 275 $^{o}$C for 60 min.




*Further annealing at 275 $^{o}$C* produces an apparent decrease in the concentration of the extended defects. Occasionally, large segmented extended defects are observed.

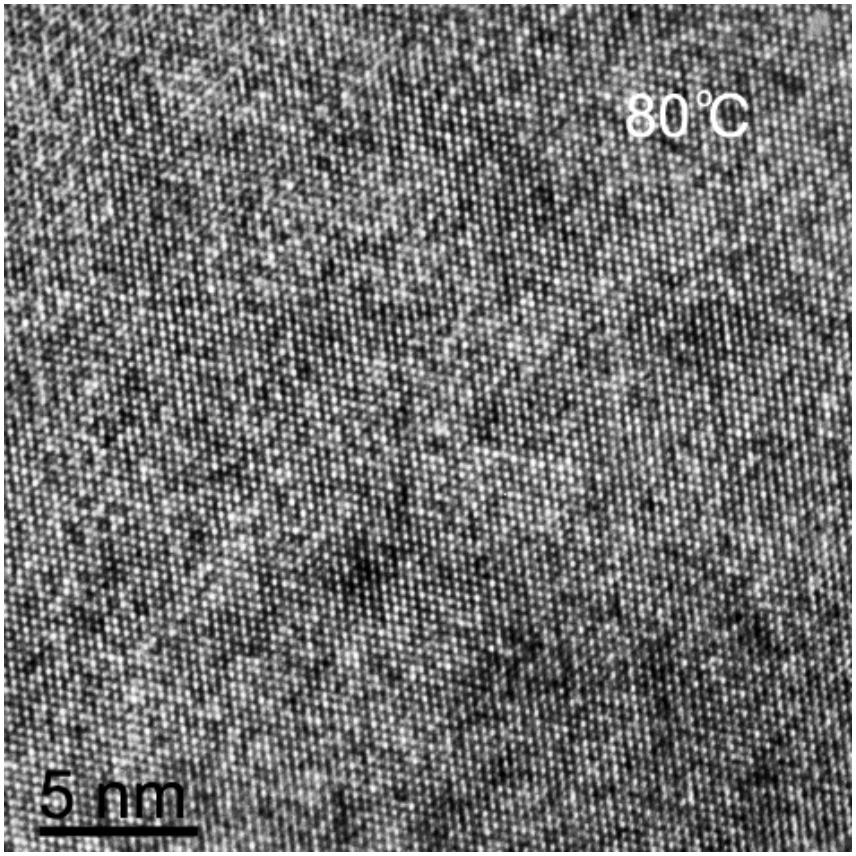
# The 27 MeV irradiated specimen



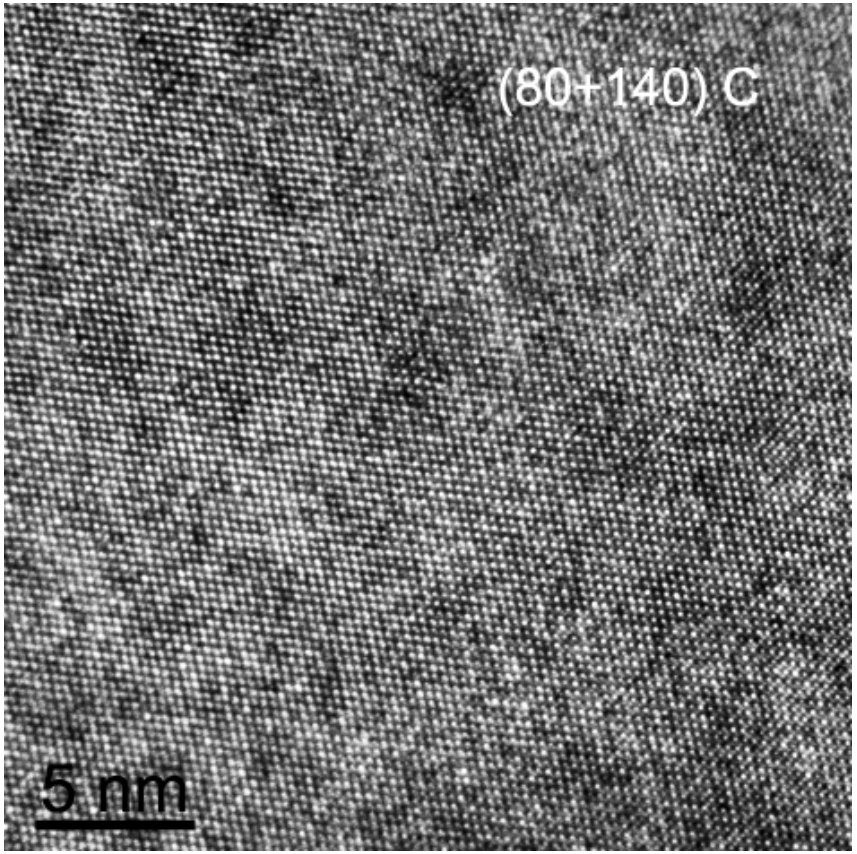

*HRTEM images of the specimen after electron irradiation*. A high density of clusters of point defects (vacancies and interstitials) appears, some grouped along the principal crystallographic directions.

The 27 MeV irradiated specimen annealed at 80 $^{o}$C for 30 min.



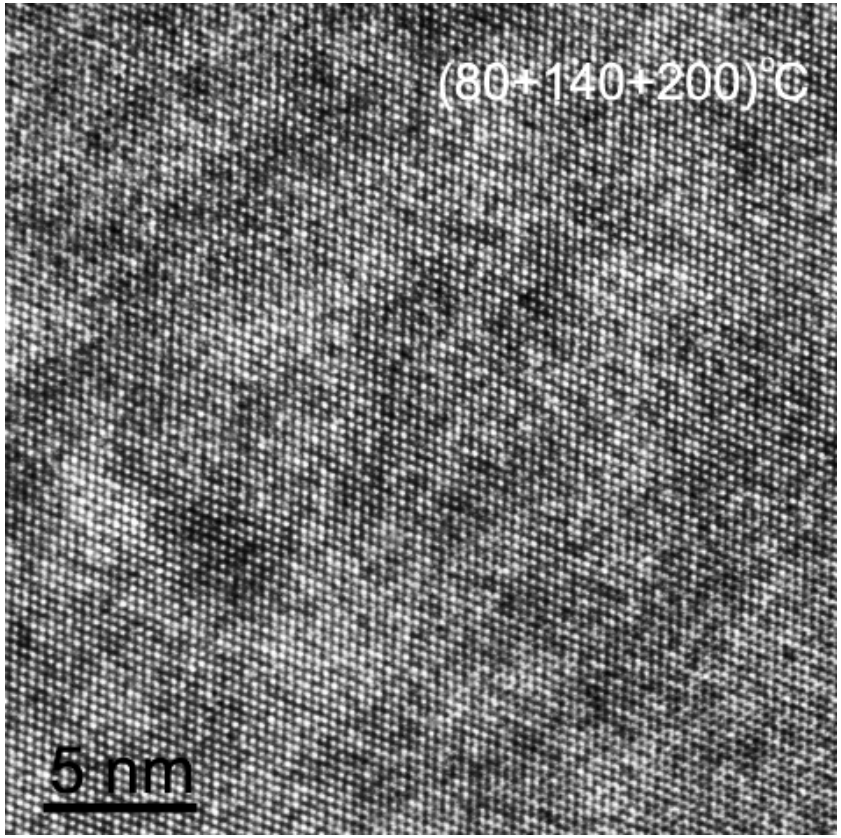

*Low temperature annealing* does not produce a significant changes in the density of the clusters of point defects except a de-grouping (migration ?) of the defect clusters.

The 27 MeV irradiated specimen annealed at 80 $^{o}$C for 30 min + 140 $^{o}$C for 30 min.



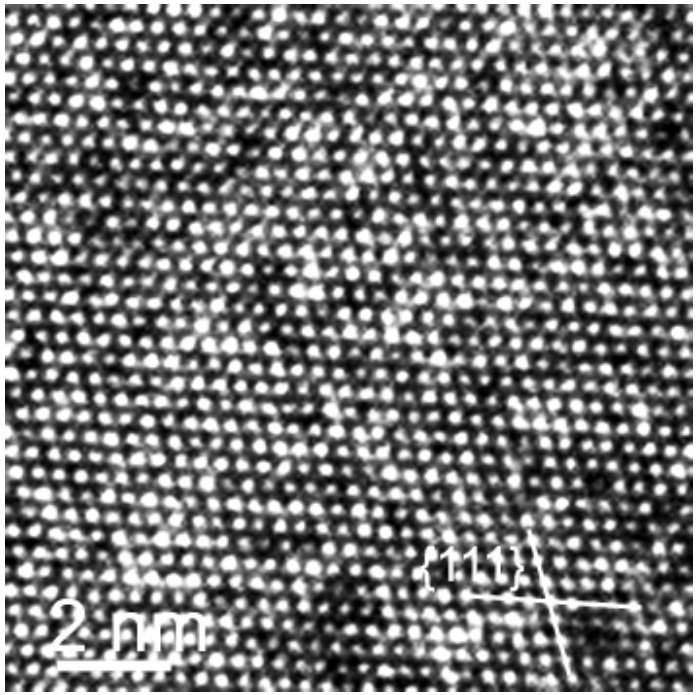

*Further annealing at 140 $^{o}$C* produces an apparent agglomeration (re-grouping) of the clusters of point defects.

The 27 MeV irradiated specimen annealed at 80 $^{o}$C for 30 min + 140 $^{o}$C for 30 min. + 200 $^{o}$C for 30 min.

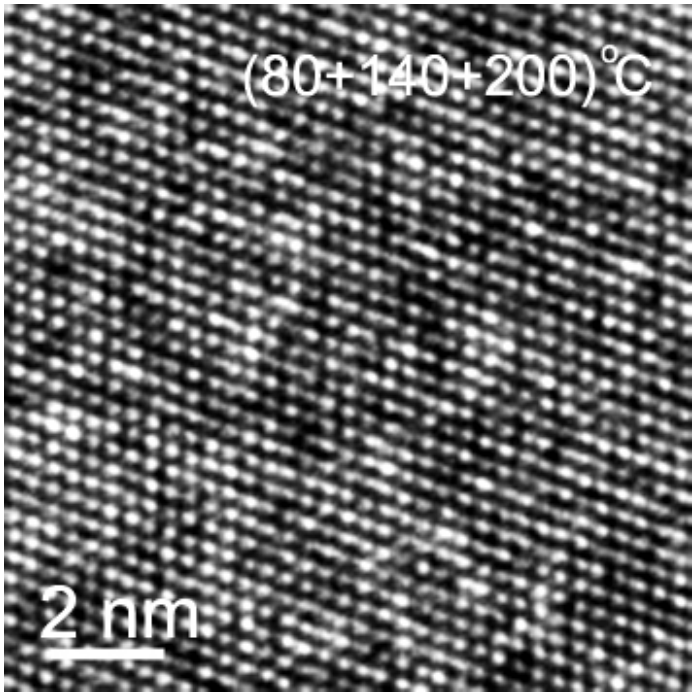



*By further annealing at 200 $^{o}$C* the agglomeration of the clusters of point defects increases.

# The effect of thermal annealing on the 27 MeV irradiated specimen




Just irradiated

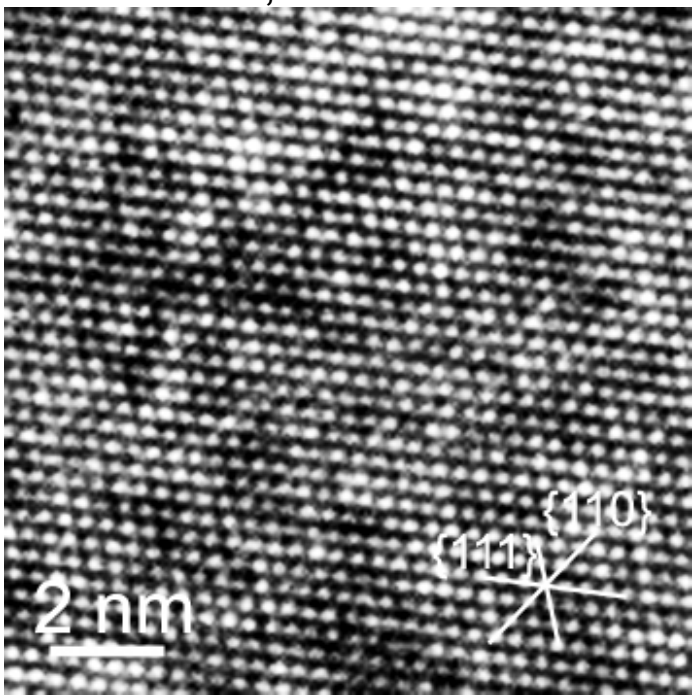



Thermal annealed

There is a tendency for the clusters of point defects to agglomerate by isochronal annealing till 200 $^{o}$C. This effect seems to take place by a migration process of the defect clusters, meaning de-grouping and re-grouping, as the temperature is increased.

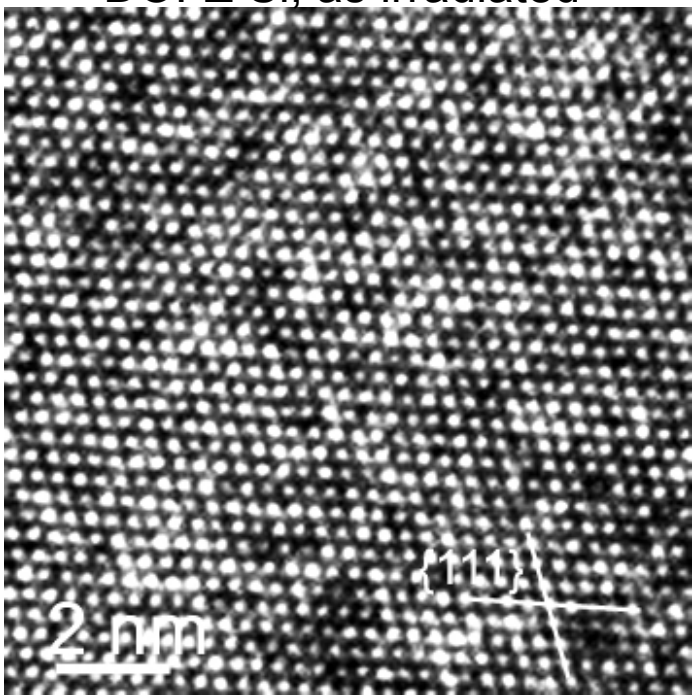
## Effects of high energy electron irradiation on FZ Si samples with and without diffused oxygen.

STFZ Si, as irradiated




15 MeV; $1 \times 10^{16}$ $cm^{-1}$

DOFZ Si, as irradiated




27 MeV; $2 \times 10^{16}$ $cm^{-2}$

Although the energy and dose for the DOFZ Si sample were higher than for the STFZ Si sample, no significant differences appeared in the density of the defect clusters introduced by electron irradiation as revealed by HRTEM.

## Conclusions

1. The HRTEM studies revealed that the high energy electron irradiation introduces in FZ Si clusters of point defects (vacancies and interstitials).
2. Regardless the presence of diffused oxygen in the Si samples (STFZ vs. DOFZ ) the clusters of point defects show similar contrast and distribution the along principal crystallographic directions.
3. The density of the introduced defect clusters, as observed in the HRTEM images, does not apparently differ, although the two samples were irradiated at different electron energies (15 MeV the STFZ, 27 MeV the DOFZ) and doses ($1x10^{16}$ $cm^{-2}$ the STFZ and $2x10^{16}$ $cm^{-2}$ the DOFZ).
4. Annealing experiments in the temperature range 80 – 275 $^{o}C$ for various periods of time produced in the STFZ Si sample extended defects such as {111} Frank dislocation loops formed by the aggregation of vacancies or {113} rod-like defects formed by the aggregation of self interstitials.
5. It has been observed that when the extended planar defects appeared, the clusters of point defects introduced by irradiation were no more visible in the surrounding regions. It looks like the Si lattice has been recovered.
6. Isochronal thermal annealing of the DOFZ Si sample in the temperature range 80 - 200 $^{o}C$ for 30 min. produces an apparent migration of the defect clusters by de-grouping at 80 $^{o}C$ and re-grouping at higher temperatures.