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TITLE: Exploring sub-10[nm] oxygen clusters in Czochralski silicon
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Abstract:
Abstract Body: Czochralski silicon is grown from quartz crucibles which contribute about 10[ppma] of interstitial oxygen to the grown crystal, which in turn acts as an ally in both strengthening silicon wafers and in precipitating to create impurity-gettering extended defects. The number density and structure of these useful defects can be affected by thermal history during crystal growth, but quantitative understanding of the structural details of oxygen clustering in the sub-10[nm] range remains elusive.

For heat treatments below 600C there are thermal donor and dislocation dipole forms, which we won't discuss here. For heat treatments above 600C, one finds[1] strained octahedra with (111) facets, and strained square platelets with {110} edges which lie on {001}. The strain comes from the volume increase when oxygen comes out of solid solution and bonds with silicon. This volume increase is also responsible for the creation of a variety of interstitial-loop secondary defects in later stages of precipitation which help with the impurity gettering mentioned above. There is also some evidence for an unstrained {001} oxygen-sheet precursor[2] or "ninja" phase, which may help explain the {001} habit of those strained platelet precipitates after further growth.

We explore the hypothesis here that oxygen clustering might be better characterized by transmission electron microscopy:

(a) in higher oxygen and vacancy (e.g. solar silicon) material as these properties can result in higher nucleation densities e.g. around 10^{12}[clusters/cc], and

(b) after thinning by mechanical instead of ion milling techniques because fresh air-exposed ion-milled silicon surfaces form 10[nm] surface oxide intrusions[3] which in practice make bulk oxide clusters in that size range difficult to recognize.

Using TEM-disc anneals (with O a direct interstitial diffuser[4]) and whole-wafer anneals, we reported earlier[5] that effects of thermal history on such small clusters can be seen in the TEM after those clusters have been increased in size by subsequent growth. Here we explore improved strategies for detection of such clusters in modern electron microscopes, in which the small number density and weak strain (along with background defects) are a much bigger problem than cluster size itself.

[1] G. Fraundorf et al. (1985) JECS Solid State Science & Technology 132:7 1701-1704.
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[4] Hartmut Bracht (June 2000) MRS Bulletin 22-27.
[5] Jamie Roberts et al. (2016) Microscopy & Microanalysis 22:S3 942-943.
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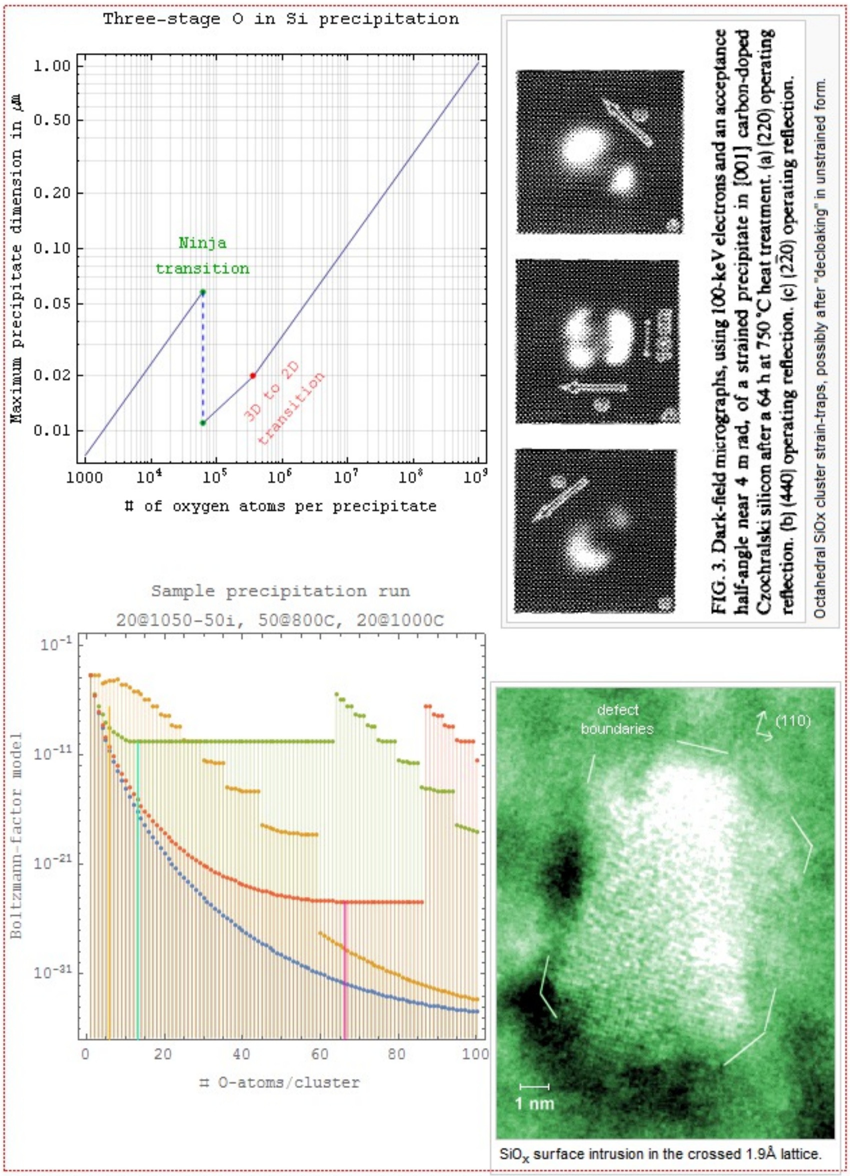

Four figures related to (top left) possible shape transitions in the clustering of oxygen in silicon, (top right) strained SiOx octahedra in the 10nm size range which on further growth might have become platelets on (001), (bottom left) Boltzmann factor model of cluster sides after freezing in at room temperature during a sequential precipitate growth anneal, and (bottom right) HRTEM image of an surface oxide intrusion following air exposure of a freshly ion milled silicon disc.