TEM study of oxygen precipitates and other defects in Czochralski-grown silicon

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Keywords: Czochralski-grown silicon, oxygen, nucleation

Precipitation of nanometre size inclusions is the process, which may substantially change physical parameters of materials. This process is crucial also in electronics, where the structural quality of semiconductor wafers is an important parameter influencing their electrical properties. Structural defects in Si ingots are created during the ingot pulling and during high-temperature technology steps. The defect nature depends on the density of self-interstitials and vacancies as well as on the density of interstitial oxygen atoms [1], the most common defect types being SiO_x precipitates, clusters of Si self-interstitials, voids, stacking faults, or combinations of them (e.g. SiO_x precipitates combined with a dislocation loop) [2].

Mastering wafer defect engineering is an important issue in semiconductor technology. Several methods are commonly used, based on empirical sequences of annealing steps. Theoretical description of defect nucleation in semiconductors can be referred to the classical theory of nucleation [3,4]. A detailed modelling of the defect nucleation and growth in Si wafers is complicated due to a large number of process parameters along the production path. Hence an extension of classical nucleation theory is required to achieve more realistic models and results.

This work is a part of a complex study of nucleation and growth of oxygen precipitates in Czochralski-grown silicon crystals. Characteristics of precipitates and associated defects in samples subjected to controlled annealing are being collected using several experimental methods, including TEM. The experimental results will be compared with the solution of nucleation equations using the method of nodal points [5,6]. The prediction and understanding of precipitation kinetics will lead to development of recipes (annealing treatments) for the growth of defects with required parameters.

Silicon wafers (diameter 150 mm, B-doped, 4–5 Ωcm) cut along (111) plane were used in this work. Two samples are compared here, differing in applied heat treatment. Parameters of annealing (temperature and time) were chosen according to the theoretical predictions. Sample Z11 underwent a three-step annealing: 8 hrs at 600°C, 4 hrs at 800°C and 12 hrs at 1000°C in a nitrogen atmosphere. This annealing sequence leads to the controlled nucleation and growth of precipitates. Sample Z12 started with annealing at 1000°C for 24 minutes and further sequence was identical to sample Z11. The first short annealing of Z12 (called Tabula rasa) removes the distribution of point defects and their clusters caused by the Czochralski growth of the Si ingot. Therefore, the subsequent nucleation and growth of defects is affected only by the process described by classical nucleation theory and not by the ingot temperature history.

TEM with EDX was used to study the samples prepared from Si wafers by grinding, dimpling and ion milling. Since the defect density is rather low, the TEM itself cannot be used for obtaining reliable statistical data on the mean defect size and defect density.

DOI: 10.3217/978-3-85125-062-6-628 , © Verlag der TU Graz 2009

However, TEM is irreplaceable in the direct visualization and characterization of individual defects. Defects observed in Z11 are: groups of tiny (around 40 nm) SiO_x precipitates hanging on dislocations (Fig. 1a), individual SiO_x precipitates (if larger, often compassed by dislocation loops) and stacking faults (Fig. 1b). In Z12, groups of SiO_x precipitates on dislocations are observed as well, some of individual SiO_x precipitates are well grown (around 400 nm) rectangular plates without surrounding dislocation loops (Fig. 2); stacking faults are not found. The comparison of defect distributions in the two samples shows, that the density of precipitates in Z11 is several times higher than in Z12. These first comparisons confirm well the results of other experimental methods and theoretical predictions.

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7. The work was supported by the Czech Science Foundation (Project No. 202/09/1013).

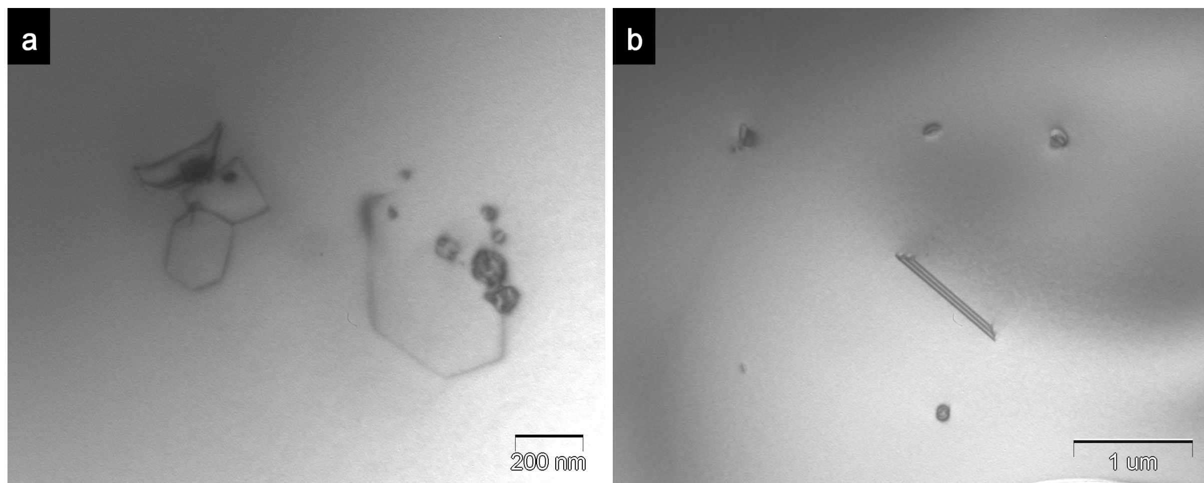

Figure 1. TEM micrographs of defects in sample Z11: dislocations with SiO_x precipitates (a) and typical dispersion of small precipitates and a stacking fault (b).

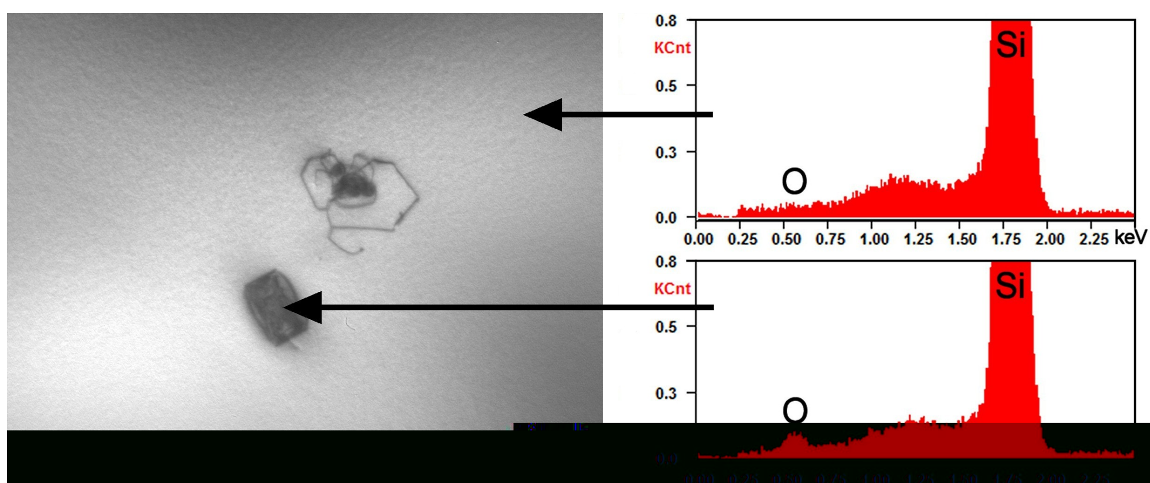

Figure 2. TEM micrograph of a grown SiO_x precipitate in sample Z12 and two EDX spectra from marked regions showing qualitatively the difference in oxygen content.

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