MICRODEFECTS IN QUENCHED CZOCHRALSKI-GROWN Si CRYSTALS

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Abstract. Optical microscopy/etch pit techniques and TEM have been used to characterize microdefects in Czochralski -grown silicon crystals after quenching from 1100°C to room temperature. The defect population is complex and consists of precipitates and rod-like defects surrounded by subsidiary precipitates. The role that oxygen may play in the formation of precipitates and rod-like defects is discussed.

1. Introduction

The investigations of microdefects in Si, formed in the process of thermal treatment are very interesting because of their influence on the electrical parameters of electronic devices.
So far, some authors[1,2] have found by TEM that prolonged annealing of Czochralski-grown silicon crystals containing oxygen concentration of about 10^{18} atoms/cm^3 causes formation of precipitates, dislocation loops, stacking faults and microprecipitate colonies on the stacking faults. For the precipitate in general it is assumed to be a form of silicon oxide[1,2].
Cullis and Katz[3] showed by transmission and scanning electron microscopy that a specific type of electrically active defects $\gamma\alpha$-FeSi_2 are present in the junction regions of silicon transistor devices.
The aim of our work was to observe the influence of quenching on the formation of microdefects in these crystals.

2. Experimental procedure

In our experiments, we used Czochralski-grown dislocation-free silicon crystals with an oxygen concentration of about 10^{18} atoms/cm^3. The crystal are in <111> orientation and doped with Boron to a resistivity of 50Ωcm.
Crystal sections of rectangular shape were cut from the crystals and were then cleaned, polished and quenched from 1100°C to room temperature by usual method.[4)]
In order to delineate microdefects in the optical microscope, after quenching of the silicon samples, we performed 10 minutes Sirtl etching[5)] in the solution, which was composed of four parts by volume of 50 gr. CrO_3 dissolved in 100 ml of de-ionized water and five parts of hidrofuluoric acid (48%). The etching is terminated by rapidly flushing away the Sirtl etch with D.I. water.
Searching fore more details about microdefects, we studied the same samples by "Simens" transmission electron microscope of 125 KeV. For these studies the samples were mechanically thinned to thickness of about 40 μm. Final thinning to the satisfactory electron transparency (about 1 μm), we carried out using chemical etching in solution of 5 parts HNO_3(65%), 1 part HF (40%) and 1 part CH_3COOH(99-100%) at room temperature.

3. Results and discussion

An optical micrograph of Sirtl etched previously quenched Czochralski-grown silicon sample is shown in fig.1. From this micrograph it is possible to distinguish the presence of two types of microdefects, which differ in size and concentration. They are called the bumps, or B shallow pits, and the hillocks H.
In our case, the distribution of hillocks is inhomogeneous and their concentration increases from the edge toward the centre of the specimen. The difference in the defect density distribution is undoubtedly related to the variation of the initial oxygen concentration across the crystal diameter.

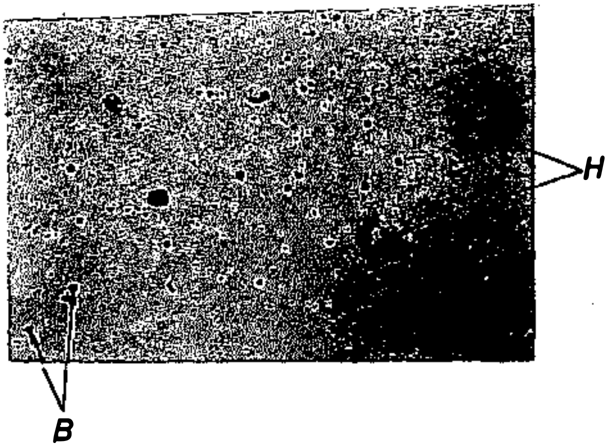

Fig.1. Optical micrograph of Sirtl etched wafer illustrating bumps, arrows B, and hillocks, arrows H.

It has been well established[6], that the oxygen concentration is much higher at the crystal center than at the edges. Some authors[6,7] associated the hilocks in Sirtl etch with defects such as bulk precipitates or stacking faults.

We obtained more details about these defects by investigation with the TEM: By the analysis of the TEM micrographs, as shown in fig.2, we noticed the precipitates ranging in size from a few tens of nanometers to several tens of micrometers. The precipitates are of different form but the larger ones have a platelike morphology. As noted above[1,2], the same form of precipitates was found in prolongedly annealed Czochralski-grown silicon crystals. For these defects it was assumed to be a form of SiO_2. In our case also there is a reason to corelate the precipitates with presence of oxygen in Czochralski-grown silicon crystals.

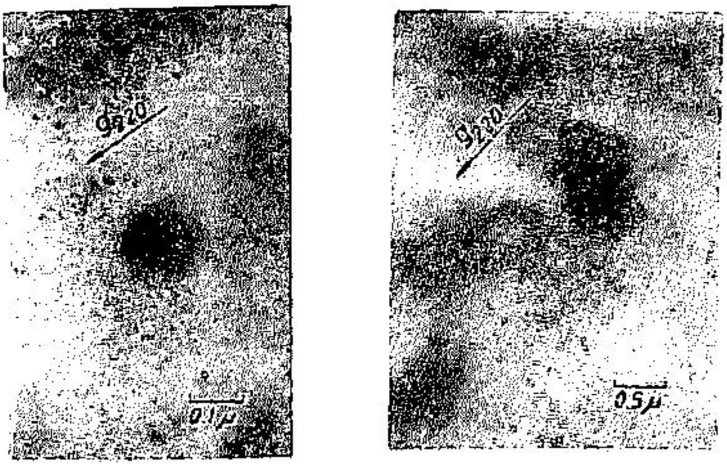

Fig.2. Transmission electron micrographs showing two types of precipitates in quenched silicon crystal.

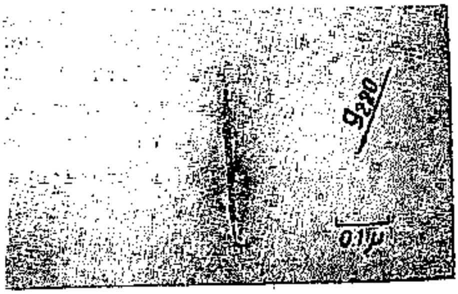

Fig.3. Transmission electron micrograph showing rod-like defect, which is surrounded by colonies of small subsidiary precipitate particles.

Another class of defects present in quenched samples are rod-like defects, as shown in fig.3. The rods have lateral dimensions of 300 nm. Such rods are usually surrounded by colonies of small precipitate particles. The geometrical configurations of these rods and precipitate colonies showed many similarities to analogous rods and colonies found by Cullis and Katz3). Because of that, the rod defects in quenched samples can be also associated with ζ_{α}-FeSi$_2$ (α-lebolite), which is metastable at room temperature8) and indeed has been observed to occur naturally as a mineral9).

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