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Non-destructive testing of silicon single crystals by positron annihilation

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Abstract. A silicon single crystal of length 25 cm and diameter 10 cm grown by the Czochralski method was cut in half; one half was examined by the Lang x-ray method after heating at 1000 °C for 16 h and the other half was examined as-grown by positron annihilation Doppler broadening. The value of the *S*-parameter was at its lowest in the region of high oxygen concentration, and at its highest in the nearly perfect lattice. The results show that silicon crystals can be tested non-destructively by positron annihilation as-grown, without requiring prolonged annealing pre-treatments.

Large single crystals of silicon are widely used in the electronics industry for the production of wafers and semiconductor devices. Nearly perfect crystals are required to produce electronic devices with high yield. Dislocations can be avoided in single crystals by making a thin neck during crystal growth. In a single crystal without dislocations, the vacancies and interstitials existing at high temperatures are not absorbed by dislocations but agglomerate, forming interstitial loops or vacancy clusters. Because there are no dislocations in the crystal, only vacancies and interstitials near the surface can be eliminated by diffusion to the surface. The vacancies and interstitials in the bulk have to agglomerate, forming interstitial clusters and vacancy clusters, or annihilate mutually by the combination of a vacancy and an interstitial. Silica crucibles are normally used to grow single crystals. During crystal growth, oxygen is introduced from the silica crucible. The oxygen hardens the crystal; further dislocations are not very easily introduced by thermal stress during the thermal treatment.

Dislocations can be detected by x-ray techniques, for example the Lang method, or by electron microscopy. However, small point defects are difficult to detect by these methods. Positron annihilation is one of the most powerful methods for detecting small point defects. It has been used very successfully in the study of point defects in metals and alloys, particularly vacancy-type defects. The situation in semiconductors is not as simple as in metals. It is not obvious that point defects trap positrons in semiconductors. This affects the trapping behaviour. Also the Fermi level changes with temperature, which affects the charge state of point defects in semiconductors.

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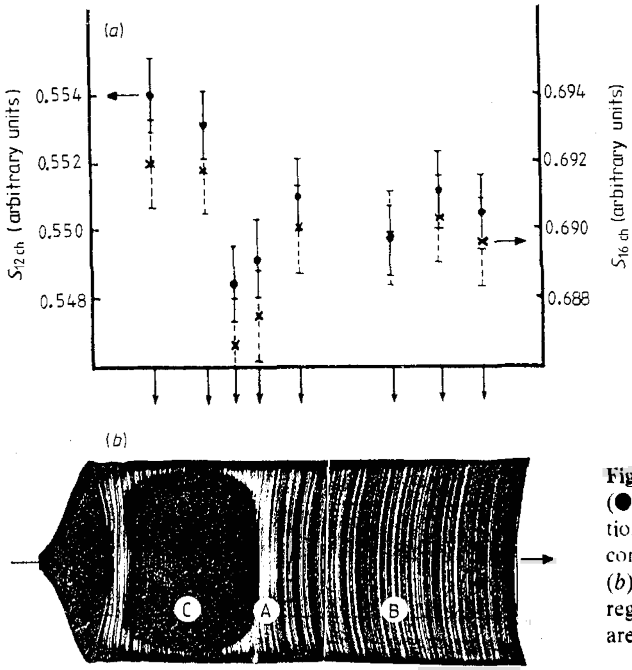

Figure 1. (*a*) Positron *S*-parameter at 77 K (●, 12 channel; ×, 16 channel) as a function of position along the length of a silicon crystal; (*b*) x-ray topograph. In part (*b*), dark areas correspond to crystal regions without dislocations and light areas to regions with oxygen precipitates.

In our experiments a single crystal of length 25 cm and diameter 10 cm was grown by the Czochralski method. The crystal was cut in half along its length, one half being examined by the Lang x-ray method, the other by Doppler-broadening positron annihilation.

It was first ensured that the crystal did not contain dislocations. The first half of the crystal was heated in argon for 16 h at 1000 °C. It was then re-examined by the Lang x-ray method. The x-ray topograph of the crystal is shown in figure 1 [1]. A widely distributed area of oxygen precipitation (marked as A) is observed. The point A is anomalous. Oxygen precipitation proceeds from A to the end of the crystal after a 16-h heat treatment at 1000 °C. This implies that oxygen precipitation is enhanced by rapid cooling from a high temperature to room temperature. The rapid cooling generates vacancy clusters and these act as nucleation centres for oxygen precipitation. Oxygen atoms then gather at the nucleation centres during the heat treatment. The area in which oxygen is reduced corresponds to the white areas of the topograph.

The other half of the as-grown crystal was sliced (2 mm thick, 20×20 mm^2) and the S-parameter at 77 K of the gamma rays emitted from the annihilation of the electron–positron pair was measured. The S-parameter is defined as $S = A/T$ where A is the number of counts in the central region of the 511 keV peak and T is the total number of counts in the 511 keV peak. The results along the length of the crystal are plotted in figure 1. The value of S is at its highest in region C, corresponding to the region of nearly perfect crystal; S is at its lowest in region A, corresponding to the region containing oxygen precipitates. Intermediate values are found in region B.

To examine the concentration of oxygen by x-ray topography, it is first necessary to heat silicon crystals in argon for a prolonged period. By positron annihilation, however, as-grown crystals can be examined non-destructively.

Reference

[1] Abe T 1985 *VLSI Electronics: Microstructure Science* vol 12, ed. N G Einspruch and H Huff (Orlando: Academic) p 3