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ELECTRON MICROSCOPICAL STUDY OF OXYGEN RELATED DEFECTS IN CZOCHRALSKI SILICON

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Résumé - Les défauts créés par un traitement à basse température du silicium Czochralski avec une teneur élevée en oxygène, sont étudiés par microscopie électronique à haute tension et à haute résolution.

Abstract - The defects formed during a low temperature treatment of Czochralski silicon with a high interstitial oxygen content are studied by both high voltage and high resolution electron microscopy.

I - INTRODUCTION

At the present day, Czochralski grown silicon crystals form the basic material for the integrated circuit industry. A large amount of investigations have been performed in the recent years concerning the influence of the oxygen content, which is always very high for this crystal growth technique, on the device performance. Both harmful and beneficial effects of its presence in silicon are reported. Among the undesired characteristics are : the formation of thermal and new donors during low temperature treatments /1/; the generation of lattice defects, which are mostly electrically active if they are present in the device region /2,3/; and the occurrence of wafer warpage /4/. On the other hand, beneficial effects are reported: wafer hardening due to reduced dislocation mobility /5/; and internal gettering of impurities by the precipitates and lattice defects outside the device region /6/.

In some previous reports /3,7-9/ the influence of a high temperature treatment in the range 1050°C - 1200°C on the nucleation and growth of lattice defects in the silicon bulk are discussed. It is seen that a low temperature preanneal in the temperature range 650°C - 900°C, combined with a high interstitial oxygen concentration results in the generation of a large number of lattice defects : stacking faults of the Frank and $\frac{1}{6}$ <114>-type, perfect dislocation loops in the {110} planes, and precipitates associated with irregular dislocation loops or with prismatic punching systems. In this paper, results are discussed on the influence of a low temperature annealing on the nucleation and growth of lattice defects, the presence of which induces the generation of the defects during subsequent high temperature treatments.

II - EXPERIMENTAL PROCEDURE

For this study, Czochralski grown silicon wafers are used, with a high interstitial oxygen concentration of 11.5 - 11.7 . 10¹⁷ atoms/cm³ (infrared-determined following the DIN-norm) and a low carbon content. The crystal is p-type (boron doped) and grown in the <001>-direction. Wafers are cut out of this crystal both orthogonal to the growth axis and inclined to it in order to obtain <111>-oriented slices. The low temperature treatments are performed in a nitrogen ambient in the temperature range 650°C - 900°C for 3h to 100 h.

After the heat treatments, the defects are studied by means of both high voltage electron microscopy (HVEM) with a Jeol 1250 kV instrument operating at 1000 kV and by high resolution electron microscopy (HREM) (Jeol 200 CX, 200 kV). For the latter technique, specimens are prepared in the <110>-direction, which is the most suitable

one for high resolution work, by means of a special cross-section thinning method, the details of which will be discussed elsewhere.

III - RESULTS AND DISCUSSION

The size and type of the defects present after the low temperature treatment, are changing as a function of the temperature. At the lowest temperatures (650°C-700°C) the defects can be characterized by means of HVEM-observations as dislocation dipoles, weak contrast rod-like defects and small precipitates (figure 1). At higher temperatures, the rod-like defects are no longer present and also the density of the dislocation dipoles decreases, so that, above 850°C the precipitates are nearly exclusively present (figure 2). The size of the precipitates increases with increasing temperature and they change from small nucleus-like contrast centers to well defined platelike precipitates, which are always accompanied by small perfect dislocation loops at the higher temperatures. Under no conditions, stacking faults are formed during the low temperature treatments.

The defect characteristics are determined with two-beam diffraction contrast experiments and also by means of high resolution imaging.

The dislocation dipoles (figure 1) are elongated in the $\langle 110 \rangle$ directions and can nearly all be characterized as having a 60°-type burgersvector $b = \frac{1}{2}\langle 011 \rangle$. Only a minority of the dipoles have a 90°-edge burgersvector. Their total lengths can go up to about 25 μm . Usually they are partitioned in small units with different 60°-burgersvectors. The change-over most probably occurs due to the interactions at small oxide precipitates. In the cross-section specimens, the dislocation dipoles can be studied end-on, and the dislocation core can be imaged with HREM. Figure 3 shows an image of an interstitial-type dislocation dipole consisting of two 60°-dislocations with opposite burgersvectors. It can be seen that the core region is free of any decoration and that the dislocations are not dissociated in partial dislocations. Similar observations are reported by Bourret et al/10/ in deformed silicon crystals, in which case however, the dislocations are always dissociated in partial dislocations.

Figure 1 also shows an example of the weak contrast rod-like defects (indicated 'b'). These have never the strong diffraction contrast which is common for dislocations. They also have their elongated dimension in the $\langle 110 \rangle$ directions and they often connect dislocation dipoles. For the image formed with the diffraction vector $g = (220)$ parallel with the line direction, perfect extinction occurs; but for all other diffraction conditions, no obvious contrast variations are observed. Therefore the displacement field can be described by $R \propto [1\bar{1}n]$ with $n > 1$ or also by $R \propto [001]$.

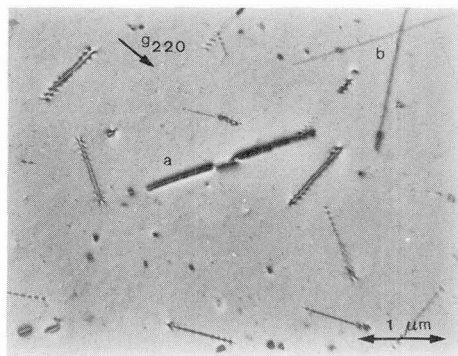


Figure 1 - The defects observed after an anneal at 700°C for 100 h : dislocation dipoles (a), weak contrast rod-like defects (b) and small precipitates.

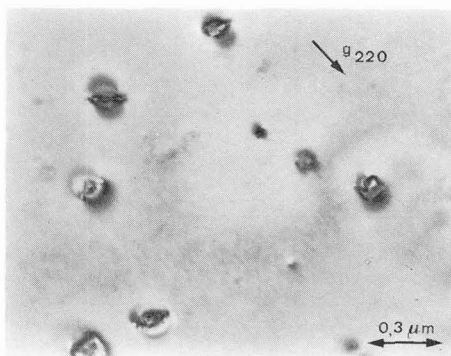


Figure 2 - Platelike precipitates and small perfect dislocation loops after a treatment at 850°C for 100 h.

This last value is often dedicated to the rod-like defects found in annealed specimens after ion implantation /11/. Due to its narrowness, no good determination of the habit plane is possible. Lambert and Dobson /11/ reported that a majority of their rod-like defects lie on the $\{111\}$ planes, but they also observed $\{100\}$ loop planes. Still a lot of discussion exists in the literature concerning the real nature of these defects. The $[001]$ -displacement vector is also accepted by Tempelhoff et al /12/ for the weak contrast defects which they observe after analogous treatments as discussed here. No good high resolution images, which would facilitate an undisputable structure determination are available as yet from these defects.

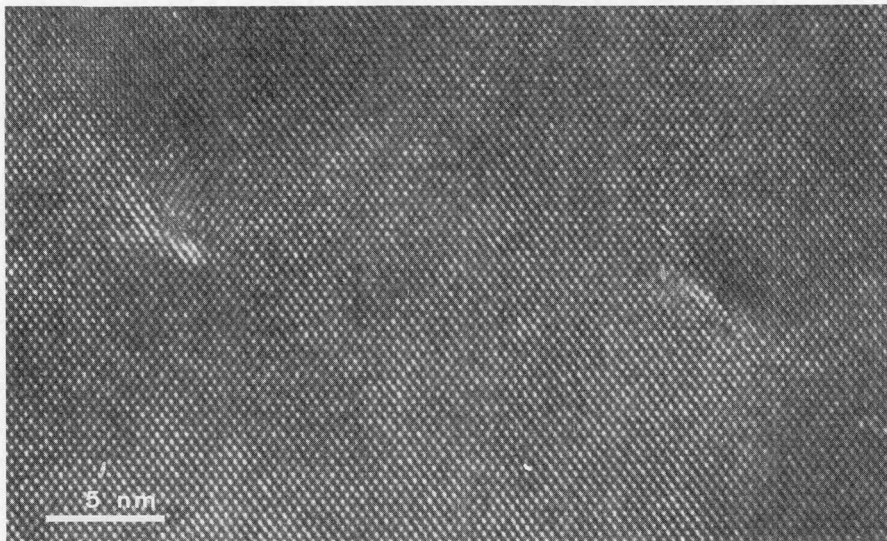


Figure 3 - HREM-image of a 60° -dislocation dipole as observed after an anneal during 15 h at 750°C .

Whereas the exact nature and atomic structure of the rod-like defects is not yet fully solved, the same can be said about their formation mechanism. A $[001]$ defect would be a high energy state and can therefore not be stable. Lambert and Dobson /11/ discuss a transition mechanism from the perfect $[001]$ defects towards perfect $\frac{1}{2}[110]$ dislocation loops via an intrinsic stacking fault $\frac{1}{6}[114]$ followed by an extrinsic stacking fault of the Frank type $\frac{1}{3}[111]$. These transition states are not present in our experiments, as no faulted defects are observed. Tan et al /13/ discuss on the basis of an atomic structure model, the formation of intermediate defect complexes (IDC) due to the aggregation of interstitials along $\langle 110 \rangle$ directions. These IDC's are assumed to be able to transform further into 60° -dislocation dipoles, 90° -edge dipoles or Frank partial dislocation loops. The required interstitials are generated in the silicon matrix due to the precipitation of oxygen in the case of the low temperature annealing experiments, or due to the annealing of the implantation damage in the case of ion implantation experiments. Transformations such as assumed in the model of Tan et al, are indeed observed in our experiments and are also reported by Tempelhoff et al /12/. However, this theoretical model needs further refinement once the real structure of the weak contrast rod-like defects is fully solved.

The third type of defects, the precipitates, are to be divided into two groups. At the lowest temperatures, small displaced areas are observed on the high resolution images (Figure 4) of about 4 nm diameter. In these areas the normal projection of the silicon lattice is locally distorted, but no extra lattice planes are visualized. This contrast behaviour is supposed to be associated with small oxide precipitates formed in the silicon lattice due to the precipitation of the supersaturated oxygen present in the wafers. The size of these small defects, agrees very well with the predictions of a homogeneous precipitation model as discussed by Inoue et al /14/.

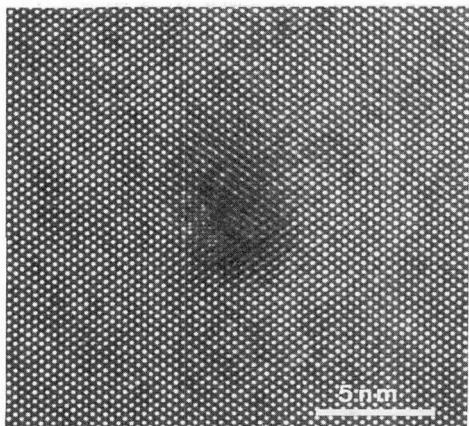


Figure 4 - Small oxide precipitate nucleus as found after a treatment at 650°C for 100 h.

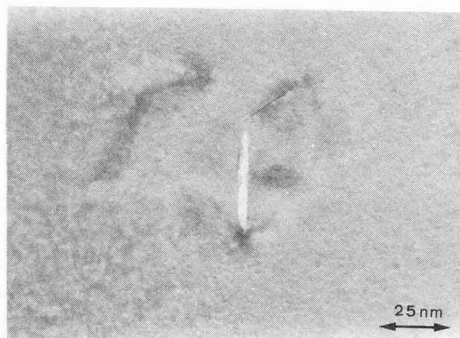


Figure 5 - Platelike precipitate and associated strain field (N_2 850°C 15 h).

At higher temperatures, these small distorted areas are only rarely observed, and they can be interpreted as the initial nucleation centers of the larger platelike precipitates, which are the dominating defects at the higher temperatures.

For temperatures above 750°C, platelike precipitates (Figures 5, 6) with their principal plane parallel to the $\{001\}$ planes occur. These consist of amorphous silicon oxide, the exact composition, however, is not known. No indications are found for the presence of crystalline silicon dioxide precipitates as reported by Matsushita /15/, although his experimental conditions are comparable with those described here. The maximal thickness of the precipitates is about 4 nm, whereas the large side can grow to more than 50 nm. These dimensions are in agreement with the values given in the study of the platelike precipitates, performed by Wada et al /16/. The surrounding silicon matrix is in a compressive state, as can be seen on the distorted lattice planes parallel with the precipitates. Therefore it is not surprising that, if they are large enough, they always generate perfect dislocation loops, which can partially reduce the existing stresses. The dislocation loops always remain pinned at the precipitates and no prismatic punching systems are observed as those found after double annealing treatments /8/.

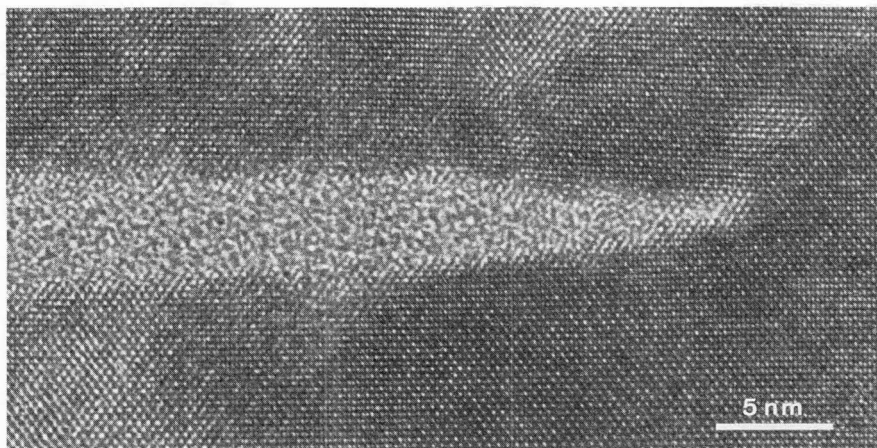


Figure 6 - Lattice imaging of an amorphous oxide precipitate observed after an anneal at 850°C during 15 h.

IV - CONCLUSION

The defects which are present after a low temperature annealing can be characterized as dislocation dipoles, weak contrast rod-like defects with presumably a $\langle 001 \rangle$ displacement field and oxide precipitates. These defects form the nucleation centers for the lattice imperfections found after a high temperature treatment during which most of the original low temperature defects are dissolved and replaced by other types.

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