Transmission electron microscopy analysis of heavily As-doped, laser, and thermally annealed layers in silicon

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The extended defects in laser activated and subsequently thermally annealed high concentration arsenic layers have been investigated in a range of surface concentrations from 2.3×10^{20} to 1.9×10^{21} As/cm^3 with transmission electron microscopy. We observe a rapid change in the density of dislocation loops with dose which is indicative of a homogeneous nucleation mechanism. The number of atoms bound by the defects is insufficient to account directly for all of the inactive arsenic. The defects lie uniformly inside the As layer up to the junction depth, which suggests that As inactive complexes are aiding the loop formation. Our results support the proposition that arsenic deactivation injects silicon interstitials. © *1995 American Institute of Physics.*

I. INTRODUCTION

Arsenic is the most commonly used dopant for creating n^+ layers in silicon. In order to get high conductivity, arsenic is incorporated into these layers in excess of its solid solubility. Subsequent thermal annealing deactivates the arsenic and gives rise to the formation of defects whose precise nature is still unknown.

Several transmission electron microscopy (TEM) observations have revealed precipitate-like defects, rod-shaped structures, and/or dislocation loops in laser and subsequently thermally annealed samples which have been doped with arsenic in excess of its electrical solubility.[1–6] Lietoila *et al.*[1] have suggested that rod-like defects may be arsenic precipitates, whereas Armigliato *et al.*[3] have reported that all defects observed by TEM cannot explain the amount of electrically inactive arsenic in their experiments. No extended defects were detected by Lietoila *et al.* for a much smaller arsenic dose. Parisini *et al.*[4] have measured the number of atoms bound by various types of interstitial-type defects at different annealing temperatures and confirmed the large discrepancy between the concentration of inactive arsenic and the concentration of atoms in observable defects. They proposed that the extended defects are formed as a result of agglomeration of silicon interstitials which are created during laser annealing. In a later work,[5] based on double-crystal x-ray diffractometry (DCD) and extended x-ray absorption fine structure analysis (EXAFS) measurements, they suggested that deactivation of arsenic is the cause of the excess interstitials. When an electron beam was used for annealing instead of a laser, similar defects were observed upon subsequent thermal treatment between 600–900 °C.[7]

On the other hand, a band of As-related precipitates and dislocation loops have been detected after solid phase epitaxy of arsenic layers doped in excess of its electrical solubility.[8–11] These defects were shown to lie at a depth corresponding to the projected range of the implant. In addition, half-loop dislocations that are located near the surface have been observed to grow during arsenic precipitate dissolution even after 72 h at 900 °C.[11] Jones *et al.*[12] have found that the elimination of end-of-range damage (category-II dislocation loops) is enhanced in the presence of a high concentration arsenic layer. Both half-loop formation and enhanced elimination of category-II dislocation loops occur when arsenic peak concentration exceeds it total solid solubility.

Understanding the mechanism of these defects is important for both defect engineering and an evaluation of the effects of As deactivation on point defects and, therefore, dopant diffusion under high concentration As layers. We report the results of TEM observations on samples which were doped with different As doses above electrical solubility and received a laser anneal to activate all of the dopant which was followed by thermal annealing. Various characteristics of the observed defects were evaluated and comparisons with published data were made.

II. EXPERIMENT

Arsenic was implanted into ⟨100⟩, 10 Ω-cm p-type silicon substrates at doses ranging from 4×10^{15} to 3.2×10^{16}/cm^2 with an energy of 35 keV. Completely active, box-shaped profiles were obtained by repeated laser pulse annealing (308 nm XeCl, 35 ns FWHM pulses, silicon melt duration 75 ns). The melt-region thickness of about 200 nm was considerably larger than the depth of the implant; thus

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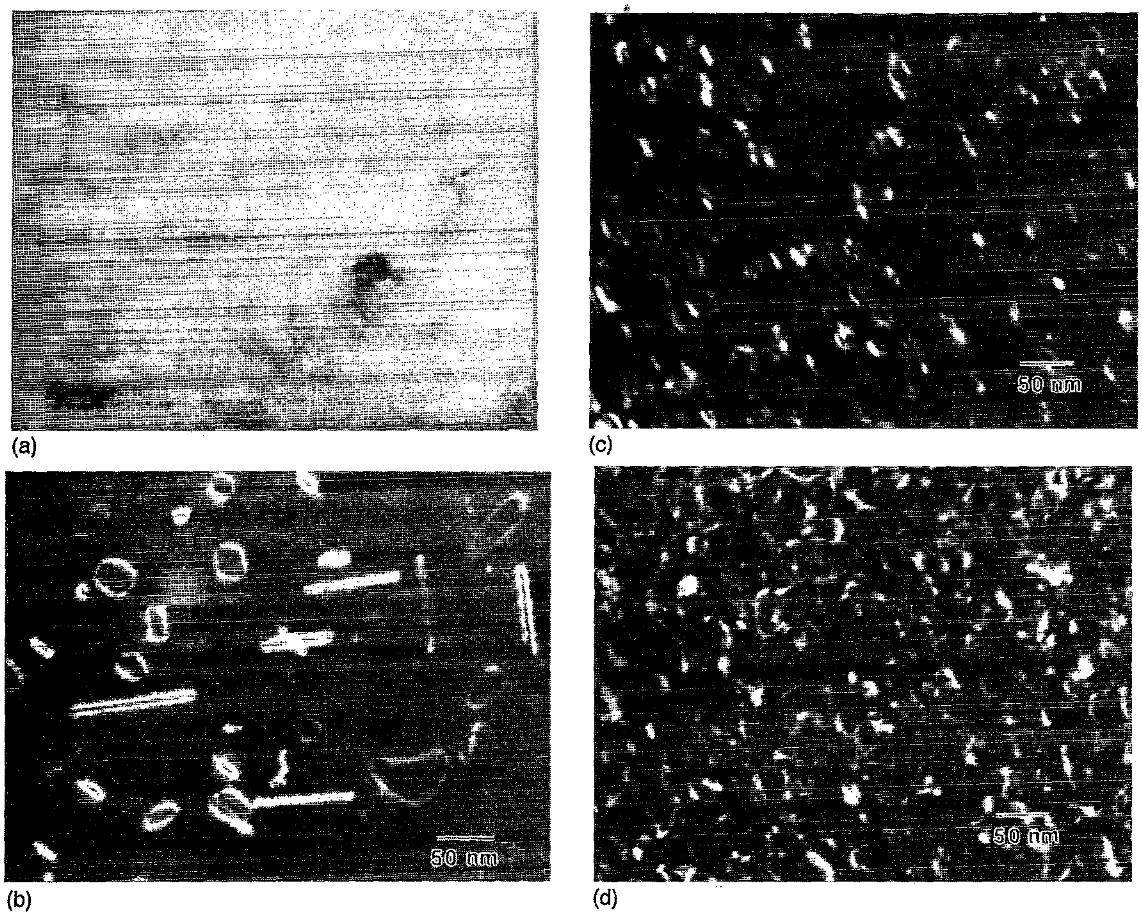

FIG. 1. Plan-view TEM micrographs of the samples annealed at 750 °C for 120 min for different doses, (a) $4\times10^{15}/cm^2$, bright field (BF), (b) $8\times10^{15}/cm^2$, weak-beam/dark-field (WBDF), (c) $1.6\times10^{16}/cm^2$, WBDF, (d) $3.2\times10^{16}/cm^2$, WBDF.

any major implant damage in the as-implanted layer was effectively annealed out. The samples then received additional rapid thermal anneals at 700 or 750 °C for durations that resulted in no appreciable diffusion.[13,14] Further experimental details about the preparation of these samples can be found in Refs. 13 and 14.

The defects were studied by transmission electron microscopy using both bright field and weak beam conditions. Both plan-view and cross-section samples were analyzed with a JEOL 200 CX electron microscope operating at 200 keV. All micrographs were taken with a g_{220} condition.

III. TEM RESULTS

The as-lased samples were completely free of any visible defects. Subsequent thermal annealing revealed a very strong dose dependence of the defect structure in the As-doped layer. This phenomenon can be seen in Fig. 1 which shows plan-view TEM micrographs of samples after a thermal treatment at 750 °C for 120 min. Density and size information about the defects is listed in Table I along with the inactive arsenic dose.

In contrast to the absence of any extended defects at the lowest dose ($4\times10^{15}/cm^2$), large dislocation loops and rod-like defects are observed upon increasing the dose by just a factor of two. In the 1.6×10^{16} and $3.2\times10^{16}/cm^2$ samples, only dislocation loops are detected. Upon increasing the As dose, the density of the dislocation loops increases. Especially, the $1.6\times10^{16}/cm^2$ sample exhibits almost fifty times more loops than the $8\times10^{15}/cm^2$ sample. The rapid change in the density of the defects between 4×10^{15} and $1.6\times10^{16}/cm^2$ is indicative of a homogeneous nucleation mechanism. The concentration of atoms bound by the loops was found to be around 30–50 times smaller than the inactive arsenic concentration and is, therefore, insufficient to directly account for most of the inactive arsenic.

Figure 2 shows the plan-view micrographs of the samples which were doped with a dose of $1.6\times10^{16}/cm^2$ and annealed at 700 °C for 15 s (short-time) and 100 min (long-time). Very dense fine particles appear after the short-time anneal whereas the long-time sample exhibits a high density of dislocation loops. Further analysis showed that the defects in the short-time sample are dislocation loops with a density of around $6\times10^{11}/cm^2$ and an average size of 90 Å. Recently, RBS measurements were made on the same samples.[15] It was demonstrated that the backscattered angular-scan spectra for Si have the same minimum in both the as-lased and short-time samples whereas the minimum yield of As increases. Furthermore, silicon minimum yield was shown to increase appreciably in the long-time sample, and both arsenic and

TABLE I. Summary of TEM observations made on the samples annealed at 750 °C for 120 min.

Dose (cm^{-2})	Surface Conc. (cm^{-3})	Defect type	Size (Å)	Density (cm^{-2})	Density of bound atoms (cm^{-2})	Inactive arsenic dose (cm^{-2})
4×10^{15}	2.3×10^{20}	None				2.2×10^{15}
8×10^{15}	4.5×10^{20}	Dislocation loops	200–1200	3.3×10^{9}	1.2×10^{14}	4.9×10^{15}
		Rod-like defects	400–2800	1.8×10^{9}		
1.6×10^{16}	9.1×10^{20}	Dislocation loops	50–350	1.3×10^{11}	$3.5–4.5\times10^{14}$	1.3×10^{16}
3.2×10^{16}	1.9×10^{21}	Dislocation loops	100–400	$>2.5\times10^{11}$	$>6\times10^{14}$	2.9×10^{16}

silicon angular-scan spectra were characteristic of a large degree of dechanneling.

Figure 3 shows the cross-section views of the samples implanted with doses of 8×10^{15} and $1.6\times10^{16}/cm^2$. For the $1.6\times10^{16}/cm^2$ case, the defects lie uniformly in a region from the surface down to a depth of about 180 nm, which coincides well with the junction depth. The uniformity of the defects in the As layer is in contradiction with an earlier TEM work where dislocation loops were observed to lie at the amorphous-crystalline interface for a similar surface concentration.[4,5] Except the intrusion of rod-like defects to a depth of 230–250 nm in the $8\times10^{15}/cm^2$ sample, the confinement of the defects to the As layer suggests that inactive As complexes are closely linked to the formation of the dislocation loops.

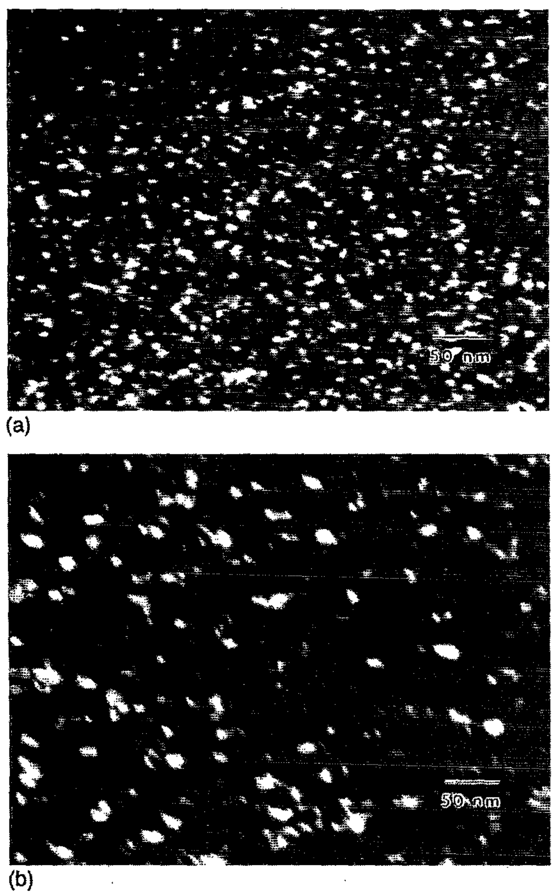

FIG. 2. Weak-beam/dark-field (WBDF) plan-view TEM micrographs of the samples implanted with 1.6×10^{16} As/cm^2 and annealed at 700 °C for (a) 15 s, (b) 100 min.

In a similar study,[5] it has been found that the dislocation loops are composed of silicon atoms. It has been suggested in the same work that arsenic clustering injects the excess silicon interstitials needed to form the loops. In a recent work, Rousseau *et al.* have observed that buried boron layers show enhanced diffusion as a result of As deactivation suggesting that As deactivation is accompanied by interstitial injection.[16] They have also found out that the enhancement decreases when As surface concentration is increased beyond $4.5\times10^{20}/cm^3$. Our TEM observations involve exactly the same samples for the 750 °C, 120 min anneal. Since the number of atoms bound by the loops increases with As surface concentration, this TEM study supports Rousseau *et al.*'s suggestion that reduced boron enhancement is caused by the dislocation loops which act as sinks for interstitials. Although the enhancement in boron diffusivity, hence the interstitial supersaturation, decreases with surface concentration, the sharp increase in the loop density between the doses of 4×10^{15} and $1.6\times10^{16}/cm^2$ can still be explained by a homogeneous nucleation mechanism (i.e., the density of the loops is a very strong function of the interstitial supersaturation), if one assumes that the initial interstitial concentration is proportional to the initial active arsenic concentration.

IV. CONCLUSION

In summary, several features of the dislocation loops formed during arsenic deactivation were investigated. First, a strong dependence of the density of the dislocation loops on initial active arsenic concentration at 750 °C was observed. This result has been explained by a mechanism in which inactive arsenic complexes injects silicon interstitials somewhat proportional to the active arsenic concentration while forming and these interstitials aggregate to form the loops. Second, the concentration of the atoms bound by the loops has a roughly linear increase with the arsenic dose, but is not

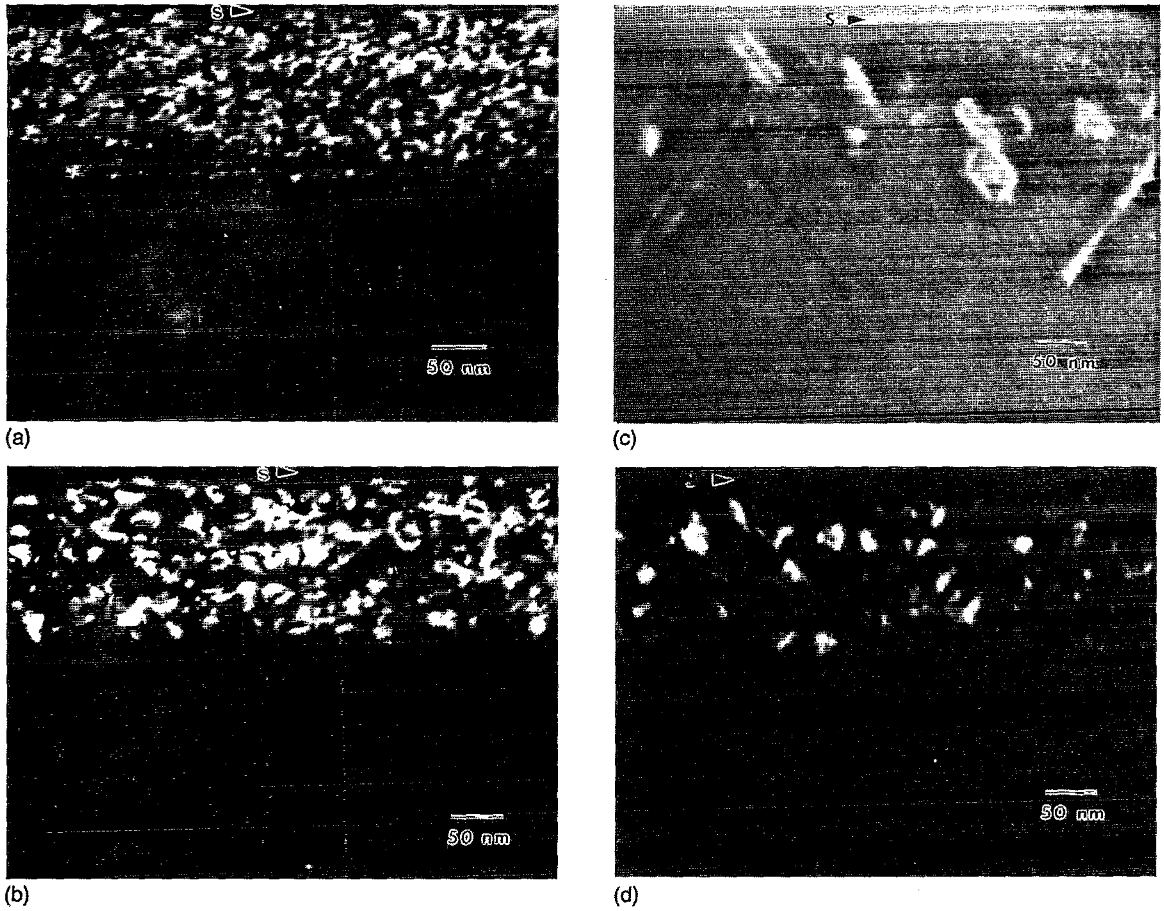

FIG. 3. Weak-beam/dark-field (WBDF) cross-section TEM micrographs of the samples implanted with doses and anneal conditions of (a) $1.6\times10^{16}/\mathrm{cm}^2$, 700 °C 15 s, (b) $1.6\times10^{16}/\mathrm{cm}^2$, 700 °C 100 min, (c) $8\times10^{15}/\mathrm{cm}^2$, 750 °C 120 min, and (d) $1.6\times10^{16}/\mathrm{cm}^2$, 750 °C 120 min.

sufficient to directly account for the inactive arsenic at all doses. Third, dislocation loops were also observed after 15 s at 700 °C and the size increased while the density decreased upon further annealing. Finally, the loops were found to be confined in the As-doped layer, which indicates that inactive arsenic is closely linked to the formation of the loops.

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