

Summary Abstract: The MBE growth of GaAs free of oval defects

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(Received 28 October 1983; accepted 8 February 1984)

PACS numbers: 68.55. + b

The oval defect has been an ubiquitous problem in GaAs epitaxial layers grown by MBE. Their reported density varies from about 100 cm^{-2} (best case) to about $10\,000\text{ cm}^{-2}$. A recent report by Calawa, *et al.*,¹ shows that oval defect formation is associated with Ga droplets which appear on the epilayer surface during growth. In this paper we report for the first time the deposition of epitaxial layers of GaAs by MBE which are *entirely* free of oval defects.

The oval-defect-free layers were grown on a 1.6 cm^2 (100) semi-insulating GaAs substrate at 550°C , at growth rates of $0.4\text{--}1.0\mu\text{/h}$. The critical factor in obtaining oval-defect-free layers was the presence of a Mg flux corresponding to a magnesium content of about 10^{19} cm^{-3} . A careful metallurgical inspection of these layers using Nomarski phase contrast microscopy at magnifications up to $1600\times$ revealed no oval defects. The hole concentrations of these layers were approximately 10^{18} cm^{-3} . Layers grown without high-level Mg doping under otherwise identical conditions had oval defect densities in the 300 cm^{-2} range.

Gallium spitting from the gallium effusion cell has long been suspected as the primary means of droplet generation. Magnesium doping should not eliminate this source, therefore we conclude that gallium spitting is not the source of gallium droplets on the substrate surface.

The remaining plausible mechanism for the formation of gallium droplets on the substrate surface is the nucleation of droplets from adsorbed gallium. When the growth surface is allowed to accumulate large quantities of gallium, many droplets form and huge oval defect concentrations result. A GaAs layer grown following exposure to about 100 monolayers of gallium had an oval defect concentration of about 10^7 cm^{-2} . The growth of oval-defect-free layers indicates that this nucleation is not spontaneous under arsenic-stable conditions. Rather, sites of surface imperfections or contamination must act as nucleating centers for gallium. However, some growth irregularities indicative of surface contamination are commonly observed and are not oval defects. Thus, only certain defects or contaminants cause the oval defect. Gallium commonly requires its oxide species to wet otherwise inert surfaces, and will not wet in vacuum certain surfaces it readily wets in the presence of atmospheric oxygen and water vapor. One might expect that a precipitate at least partly composed of gallium oxides would serve as an ideal site to nucleate a gallium droplet.

Mg is a *p*-type dopant in MBE GaAs that is known to have a lower electrical activity than is expected from the incident flux. The model most consistent with available data indicates that the reduced activity is due to the incorporation of an electrically inactive species of Mg; that species is believed

to be MgO. This indicates the existence of oxygen-containing species that readily form MgO in the presence of Mg.

Ga_2O (gallium suboxide) is an oxide species observable in the gallium cell's effuse. Intentionally increasing the Ga_2O flux from the Ga effusion cell has been shown³ to markedly increase oval defect densities. Ga_2O_3 is formed by the disproportionation of the suboxide. It is believed that this forms oxide precipitates that nucleate gallium droplet formation, thus forming the precursor of the oval defect. Moreover, the disproportionation reaction yields unbonded Ga atoms that may trigger the Ga droplet nucleation. In the presence of large fluxes of magnesium, the gallium oxides are reduced on the sample surface, forming MgO. MgO is not wet by Ga in vacuum, thus it is unlikely that macroscopic gallium droplets will be nucleated by MgO.

We performed experiments to enhance the formation of Ga_2O_3 precipitates in an attempt to induce Ga droplet formation and thereby increase the oval defect density. We used an independent source of Ga_2O , consisting of an effusion cell charged with both Ga and Ga_2O_3 . This allowed stable control of the Ga_2O flux independent of the gallium source used for growth. A Ga_2O pressure of about 5×10^{-9} Torr at the substrate resulted in an oval defect density of $10^5\text{--}10^6\text{ cm}^{-2}$. Optical properties were affected as well. The near-band-edge photoluminescence intensity at 2 K was reduced approximately tenfold compared to control specimens grown with the Ga_2O source shuttered. These control layers exhibited oval defect densities of $200\text{--}500\text{ cm}^{-2}$.

In conclusion, the role of gallium droplets in the formation of oval defects is supported. The elimination of oval defects through the use of magnesium doping indicates that these Ga droplets do not originate externally to the surface, i.e., at the gallium effusion cell. Rather, it is believed that nucleating centers on the sample surface support Ga droplet formation. It is found that a sufficient quantity of magnesium disrupts this mechanism.

The reactivity of magnesium and its observed doping behavior indicates that at least some magnesium is incorporated as MgO (at the 10^{19} cm^{-3} level, about 90%). This, in turn, suggests that in the absence of sufficient quantities of Mg, Ga_2O_3 is incorporated as precipitates. Ga_2O_3 precipitates can nucleate Ga droplet formation: Enhancing oxide incorporation with an additional Ga_2O source drastically increases the oval defect concentration. Gallium oxide species are eliminated via reactions with Mg fluxes. For properly prepared surfaces free from other nucleating centers, this results in oval-defect-free epilayers.

Our results and interpretation are consistent with the accepted origins of the oval defect¹ and the observed doping

behavior of Mg.⁴ They suggest that the suppression of residual oxide sources during MBE growth should allow the growth of higher quality crystals free of oval defects even without the use of Mg doping, consistent with earlier predictions of effects of volatile oxide impurities on defect formation during MBE growth.²

¹A. R. Calawa, G. M. Metze, and M. J. Manfra, presented at the Electronic Materials Conference, Burlington, Vermont, 1983.

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