Effects of substrate preparation conditions on GaAs oval defects grown by molecular beam epitaxy

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Effects of substrate preparation conditions, i.e., wet chemical and ultrahigh vacuum cleaning preparations, on GaAs oval defects grown by molecular beam epitaxy (MBE) were investigated. It is found that, with our MBE system, the presence of the smaller (< 10 µm) oval shaped defects without macroscopic core particulates can be ascribed to surface microscopic contaminations. Most of the other remaining larger (> 10 µm) oval defects with core particulates observed on 1-µm-thick GaAs MBE layers are attributed to surface macroscopic contaminations. The total density is reduced to 300 cm⁻² without significantly modifying the growth cell parameters.

The macroscopic surface defects¹-⁶ which reside on GaAs epilayers grown by molecular beam epitaxy (MBE) have been an ubiquitous problem for practical applications. Several studies have indicated that the most common type of surface morphological defect is the oval shaped defect, the so-called "oval defect", oriented in the (011) direction with the density range from 10² to 10⁵ cm⁻². Furthermore, several kinds of defects among the oval type have been reported. Their origins were investigated by a number of researchers to reduce their density. Experimentally the defect formation was related to the substrate preparation,³⁻⁶ the Ga source cell (Ga spitting⁴,⁵ and Ga oxides⁴), and the As source cell.³ However, the factors which cause the major oval defects are still controversial, and it is likely that there is not just a single cause but several, depending on the particular growth conditions employed. There are various kinds of oval defects, and the densities appear to vary greatly in a complex way with the growth conditions. Because of the density variations by many reasons, it seems, in general, to be difficult to correlate the defects and their origins. Therefore, we consider it very important that we should classify the oval defects and then investigate the trends when discussing the causes of the oval defects. Figure 1 shows the two representative kinds of oval defects which are of primary importance. These are the smaller size (< 10 µm) oval defect without macroscopic core particulates (called type α hereafter) and the larger (> 10 µm) oval shaped hillocks with a nucleus at their center (type β). The density of the α-type defect was typically >10⁴ cm⁻² when observed, while that of the β type was distributed between 10² and 10⁴ cm⁻².

In this letter, we study effects of substrate preparation conditions on the two types of oval defects observed on 1-µm-thick GaAs layers grown by MBE. Using our MBE system, we present experimental evidence that surface microscopic contaminations (probably carbon) due to the contaminants in the cleaning solutions and/or in the vacuum can cause the α-type oval defect. The β-type oval defect is attributed to the macroscopic contaminations on the substrate surface. With elimination of both microscopic and macroscopic contaminations through careful substrate preparations, we show that the total defect density is reduced to about 300 cm⁻² for 1-µm-thick GaAs layers without significantly modifying the epitaxial source parameters.

GaAs layers (1 µm) were grown on (100) liquid encapsulated Czochralski semi-insulating substrates with an etch pit density of 10⁵ cm⁻² in a Varian MBE-Gen II system. The growth rate was kept constant at 1 µm per hour at the substrate temperature of 630 °C. Substrate preparation procedures were as follows. The wafers were first degreased in trichloroethylene, acetone, and isopropyl alcohol, and then

FIG. 1. Nomarski phase contrast micrographs of GaAs oval defects: (a) α type without macroscopic core particulates and (b) β type with macroscopic core particulates.

FIG. 2. (a) and (b) are Nomarski phase contrast micrographs of a 1-µm GaAs MBE epilayer on the substrate prepared under optimized preparation conditions.
etched in H$_2$SO$_4$ at room temperature and in 5:1:1 (H$_2$SO$_4$; H$_2$O$_2$;H$_2$O) etchants at 60 °C. After the etching, the wafers were rinsed in de-ionized water or distilled de-ionized water and then blown dry with a filtered nitrogen gun in a clean room. The clean wafers were immediately mounted on Mo blocks with In melt before being loaded in the sample loading chamber. The substrates were preheated in the preparation room (the base pressure ~4 x 10$^{-11}$ Torr) at 400 °C to eliminate air bubbles contained in the In melt and to outgas the sample and holder assembly to a pressure below 5 x 10$^{-9}$ Torr. Before the growth, the substrates were thermally cleaned at 700 °C to remove the surface oxides in the growth chamber under As$_4$ pressures of ~10$^{-3}$ Torr. After the growth, the epilayers were examined under a Nomarski optical microscope at magnifications up to ×2000.

Figure 2 shows the micrographs of the highest quality 1-$\mu$m GaAs epilayers. For this growth, the substrate was prepared using distilled de-ionized water, mounted, loaded, and grown as quickly as possible to avoid contaminations after etching. As shown in Fig. 2(a), typically, only two or at most three $\beta$ oval defects were present in the photograph at the magnification of ×100. This gives an averaged area defect density of ~300 cm$^{-2}$. It is important to point out that the $\alpha$ oval defect was not observed on the epilayer at all. This is one of the reasons why we could reduce the total defect density repeatedly below 10$^3$ cm$^{-2}$. When the substrate was prepared using the ordinary de-ionized water (the difference is simply the use of ordinary instead of distilled de-ionized water), we often observed a significant increase of the $\alpha$ oval defect with a density around 10$^4$ cm$^{-2}$ as shown in Fig. 1(a). In addition, we also observed an increase of the $\beta$ oval defect density. These results indicate that both microscopic and macroscopic contaminations on the substrate surface resulted in the formation of the major oval defects but in different ways.

Elimination of the $\alpha$ oval defect with the use of the distilled de-ionized water might indicate that the electrically neutral contaminants in water caused microscopic contaminations, and resulted in the $\alpha$-type oval defect. In order to examine the hypothesis that the microscopic contamination is the origin of the $\alpha$-type defect, another experiment was conducted as follows. The GaAs substrate prepared using distilled water was once heat cleaned in the growth chamber to 700 °C as is the usual procedure before growth to remove the surface oxide passivation layer under As$_4$ molecules. The surface is considered to be active to the adsorbates such as CO in the vacuum chamber because of the unsaturated dangling bonds. Then, we left it for ten days to contaminate the surface intentionally. After that, the 1-$\mu$m-thick GaAs epilayer was grown on the contaminated substrate. The result of microscope surface observations is shown in Fig. 3. A fairly uniform distribution of the $\alpha$-type oval defect oriented in the (011) direction is clearly seen. This indicates that the microscopic contaminations from either the cleaning solution or the vacuum residues cause the $\alpha$ oval defect. The density was usually very high (~10$^4$ cm$^{-2}$).

The other remaining major oval defect is the $\beta$ type with the minimum density of 300 cm$^{-2}$. Figure 4 gives unambiguous experimental evidence that the $\beta$ oval defect can
also be due to the surface macroscopic contaminants as also studied by Weng et al. Near the periphery of the wafer the contaminants were easily condensed when the substrate was blown dry. Sometimes we observed a clear border curve as in Fig. 4(a) for the distribution of the $\beta$ oval defects. Also shown in Fig. 4(b) is a distribution of the $\beta$ oval defects in a line which accidentally occurred when loading the wafer. These distributions of the $\beta$ oval defects are not caused by Ga spitting. All of the above experimental results demonstrate that the substrate surface preparations can be important in causing the formation of the major oval defects.

In summary, we have investigated effects of substrate preparations on two major oval defects and their density on 1-$\mu$m GaAs epilayers grown by molecular beam epitaxy. It is shown with our MBE system that the relatively smaller, dense oval defects without macroscopic core particulates are attributed to the microscopic contaminants. With complete elimination of these small oval defects by careful and optimized substrate preparation, the total defect density is reduced to 300 cm$^{-2}$, due to the remaining oval defects with core particulates.