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Nearly-Dislocation-Free, Semi-Insulating GaAs Grown in B_2O_3 Encapsulant

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This paper describes the growth of dislocation-free GaAs crystals by the Fully Encapsulated Czochralski (FEC) technique in combination with the doping of the isovalent indium element. It is demonstrated that dislocation-free, semi-insulating crystals 25-30 mm in diameter can be successfully obtained, containing only a few hundred straight and axial <100> dislocations propagating from the seed crystal. This is mainly attributed to the reduced arsenic evaporation from the crystal surface, provided by the FEC technique. Nearly-dislocation-free crystals 50 mm in diameter are also realizable with this method.

1. Introduction

It has been pointed out that dislocations in GaAs crystals grown by the Liquid Encapsulated Czochralski (LEC) technique severely affect FET performance.[1)] Thus, the elimination of dislocations would help to realize high performance GaAs LSIs.

The high density of dislocations in LEC GaAs crystals is mainly due to the propagation and multiplication of dislocations from the seed crystal as well as to newly generated dislocations at the crystal periphery. The dislocations from the seed crystal can be effectively reduced by a necking procedure. On the other hand, it is very difficult to suppress the generation of dislocations at the crystal periphery for the following reasons. The conventional LEC technique exposes a grown crystal to a high temperature gas atmosphere for a long period of time during the growth processing. This results in a degraded crystal surface due to arsenic evaporation from the crystal, which acts as the origin of dislocation generation. Worse yet, this arsenic evaporation increases still more when the ambient temperature gradient is decreased in order to reduce thermal stress which causes the multiplication of dislocations.

In a previous paper,[2)] to overcome this problem, we presented a modified LEC method in which the crystal was placed in a thick B_2O_3 encapsulant throughout the growth process. Dislocations near the crystal periphery were effectively reduced owing to a reduction in arsenic evaporation from the crystal surface. We refer to this technique as the Fully Encapsulated Czochralski (FEC) method, in contrast to the conventional LEC method.

It is well known that impurity hardening is effective in suppressing the propagation and multiplication of dislocations caused by thermal stress. Recently, it has been reported by several groups[3-5)] that dislocations can be effectively reduced by the doping of the isovalent indium element. Jacob et al.[4)] have obtained nearly dislocation free crystal 20 mm in diameter. However, they have not reported the detailed profiles of dislocation density across the wafer including the degraded peripheral part caused by arsenic evaporation. In our investigation, we were not able to suppress the generation of high density dislocations at the periphery by means of the conventional LEC method combined with indium doping at a concentration of $5\text{-}10\times10^{19}$ atoms/cm^3. Moreover, it was recognized that the high density of dislocations observed as linear arrays aligned along the easy <110> glide directions extended further and reached the central region of the wafer when thermal stress became larger with an increase in crystal diameter.

In the present paper, the FEC method in

combination with indium doping has been applied to the growth of dislocation-free crystals. It has been demonstrated that dislocation generation at the crystal periphery has been effectively suppressed and dislocation free crystals 25-30 mm in diameter (excluding several hundred straight and axial <100> dislocations propagating from the seed crystal) have been obtained. Moreover, the application of the present method to the growth of large sized crystals 50 mm in diameter has been investigated.

2. Experimental

Crystals were grown by the FEC technique from a melt of about 500g containing 4.5×10^{20} atoms/cm^3 indium, in-situ synthesized in a pyrolytic BN crucible 100 mm in diameter. Figure 1 shows a schematic drawing of the setup for the FEC technique compared with that for the conventional LEC technique. A separately controlled sub-heater was installed so as to optimize the temperature gradient in the thick B_2O_3 encapsulant and to ensure the crystal pulling procedure. To confirm the advantage of the FEC technique, several crystals were also grown by the conventional LEC technique. The thickness of the B_2O_3 encapsulant used in the present experiments was about 50 mm for the FEC method and 15 mm for the conventional LEC technique. The temperature gradient in the B_2O_3 encapsulant was controlled equally for both methods, and was about 50 °C/cm. The crystals were grown in the <100> direction with a 5-10 mm/hr pulling rate. The necking procedure was used for all crystal growths. Crystal diameter and length were about 25-30 mm and 50-60 mm, respectively. The atmospheric pressure was 8 kg/cm^2 of argon.

Wafers cut from the grown crystals were subjected to investigation of dislocations by molten KOH etching and measurements of electrical properties by the Van der Pauw method. Moreover, the levels of doped indium in the crystals were measured using Secondary Ion Mass Spectrometry (SIMS) calibrated by Inductively Coupled Plasma Emission Spectrometry (ICP).

3. Results and Discussion

A typical crystal grown by the present method is shown in Fig. 2. The as-grown surface of the crystals always exhibited a metallic surface which is a characteristic of the crystals grown by the FEC method. This is due to the reduced arsenic evaporation from the crystal surface. On the other hand, the surface of the crystals grown by the conventional LEC method was rough and matted.

The measured indium concentration in the grown crystals was about 5×10^{19} atoms/cm^3 at a solidified fraction g = 0.2. Hall measurements confirmed that the electrical properties of the as-grown crystals were very similar to those obtained for normal undoped semi-insulating GaAs, i.e., a resistivity of $5\text{-}10\times10^7\,\Omega$cm, and a mobility of 4000-5000 cm^2/V·sec at room temperature.

Figure 3 shows photographs of a wafer approximately 25 mm in diameter after KOH etching. Although axial <100> dislocations from the seed crystal are observed in the central core as shown in Fig. 3 (b), other regions in the

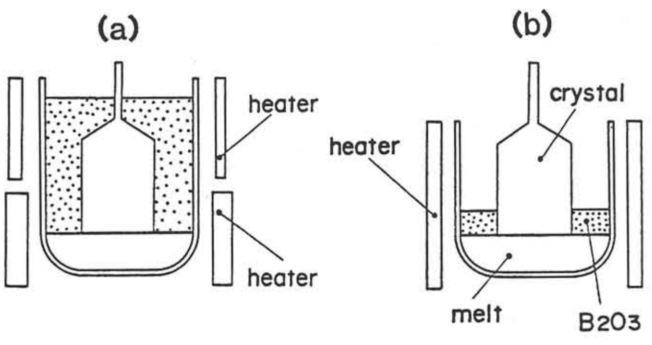

Fig. 1 Schematic drawing of the experimental setup. (a) FEC method. (b) Conventional LEC method.

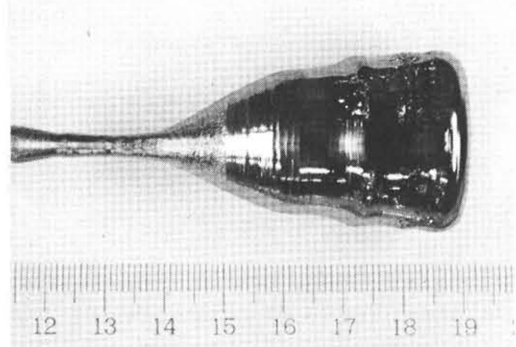

Fig. 2 Photograph of a crystal grown by the FEC technique in combination with indium doping. Crystal diameter and length are about 25 and 50 mm, respectively.

wafer are completely dislocation-free including the periphery of the wafer as shown in Fig. 3 (c). On the other hand, in crystals grown by the conventional LEC method and doped with indium, high density dislocations were observed, in addition to the dislocations from the seed, as apparent from a photograph of the wafer 25 mm in diameter shown in Fig. 4. The peripheral part of the wafer was extremely degraded due to arsenic evaporation from the crystal surface and, as a result, contained many gallium droplets around which a cloud of dislocations generated. These gallium droplets were often observed in the interior as well as the peripheral part of wafers. They were introduced at the periphery of the seed end crystal and then propagated downward along the growth axis during the growth processing. In the peripheral part of wafers, moreover, high density dislocations aligned along the easy <110> glide directions were observed.

Figure 5 shows radial variations in dislocation densities for FEC and conventional LEC grown wafers with indium doping. For the FEC grown wafer, as described earlier, dislocations were observed only in the central 2 mmϕ region of the wafer and their density was about 10^4 /cm^2. On the other hand, the conventional LEC grown wafer showed W-shaped variations in dislocation densities which were high in the central and

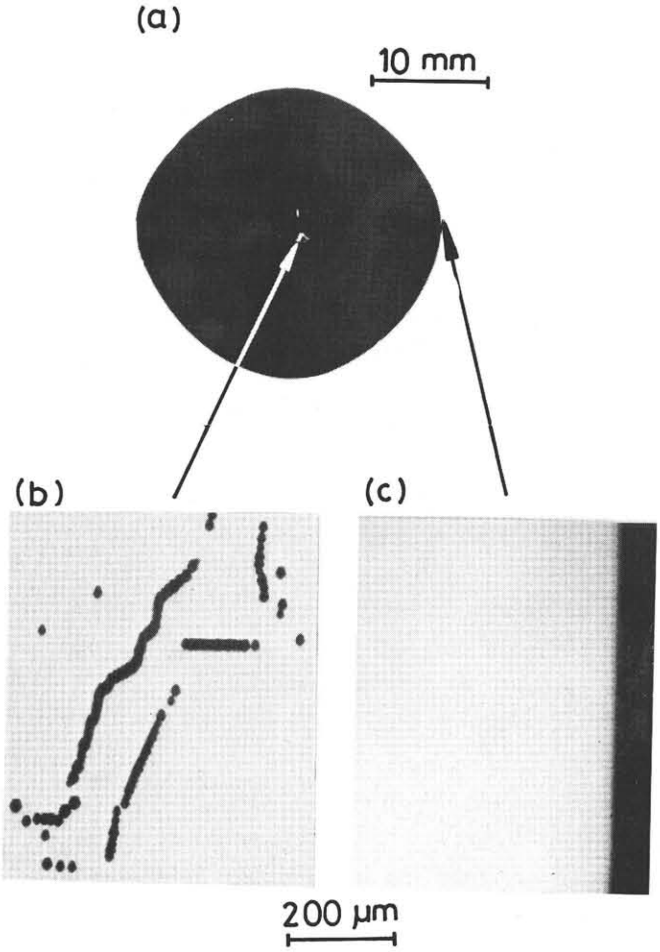

Fig. 3 (a) Photograph of a wafer cut from the crystal grown by the FEC technique with indium doping after KOH etching. White portions in the central core on the wafer correspond to dislocations from the seed crystal. (b) Enlarged photograph of the central region indicated in (a). (c) Enlarged photograph of the peripheral region indicated in (a).

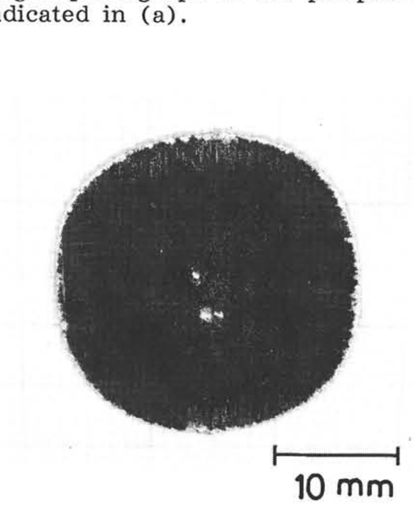

Fig. 4 Photograph of a wafer cut from the crystal grown by the conventional LEC technique with indium doping after KOH etching. White regions on the wafer correspond to dislocations.

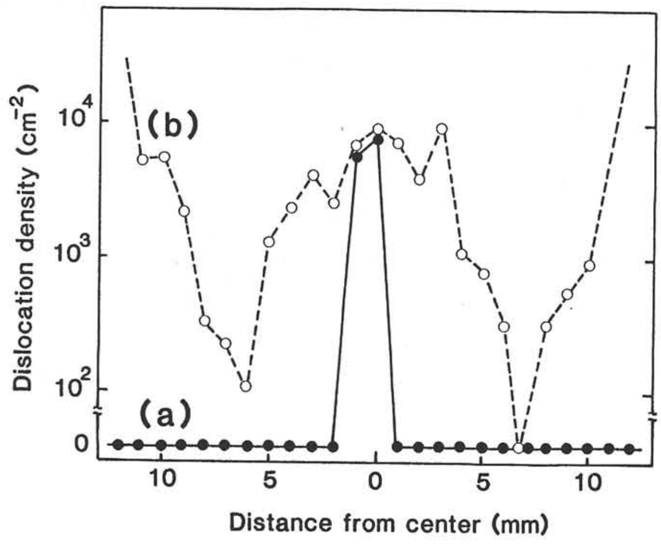

Fig. 5 Radial variations in dislocation densities measured in the <110> direction across the wafer. (a) Wafer cut from the crystal grown by the FEC technique with indium doping. (b) Wafer cut from the crystal grown by the conventional LEC technique with indium doping.

peripheral regions. High dislocation densities at the peripheral part of the wafer are apparently responsible for newly generated dislocations due to the degraded crystal surface described earlier. And the high dislocation densities in the central region of the wafer are attributed to the dislocations propagating from the seed crystal because of insufficient necking procedures. The W-shaped radial variation in dislocation densities presented here is very similar to variations normally observed in undoped LEC grown crystals, although the dislocation densities in the middle region are extremely reduced and a dislocation-free area is partly observed. However, it should be emphasized that, as evident from the present considerations on dislocation generation, the W-shaped profile of dislocation density presented here cannot be directly attributed to the radial profile of thermal stress in the growing crystal [6].

From the present experimental results, it is apparent that the FEC technique in combination with indium doping can effectively suppress dislocation generation at the crystal periphery and makes it possible to obtain larger diameter dislocation-free crystals. This is mainly attributed to the reduced arsenic evaporation from the crystal surface provided by our FEC method in addition to the hardening effects of indium doping, because the reduction in arsenic evaporation from the crystal surface is an essential factor to increase a critical resolved shear stress for dislocation generation to a required value.

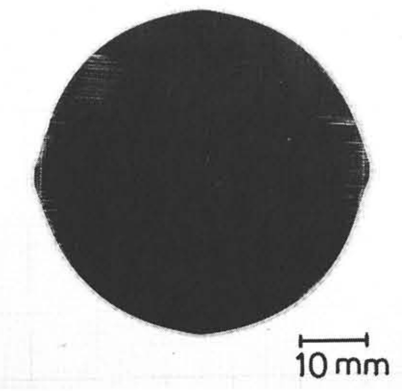

Fig. 6 Photograph of a typical wafer, cut from the 50 mm diameter crystal grown by the FEC technique with indium doping after KOH etching.

The present FEC technique employing indium doping was also applied to the growth of crystals 50 mm in diameter. Figure 6 shows a photograph of the wafer, cut from the grown crystal after KOH etching. With the FEC technique, no gallium droplets are introduced and the dislocation-free region extends to the periphery of the wafer although dislocations aligned along <110> directions are partly observed.

4. Summary

The Fully Encapsulated Czochralski (FEC) technique including doping with the isovalent indium element was applied to the growth of dislocation-free crystals. It was demonstrated that dislocation generation at the crystal periphery was effectively suppressed. This is primarily due to reduced arsenic evaporation from the crystal surface. As a result, dislocation-free, semi-insulating crystals 25-30 mm in diameter were successfully grown, which contained only dislocations propagating from the seed. It was also confirmed that, nearly dislocation-free 50 mm diameter crystals can be obtained by the present method.

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