C—2—1 (Invited)

Growth of Undoped Semi-insulating GaAs Single Crystals

T.Fukuda

Optoelectronics Joint Research Laboratory

1333 Kamikodanaka, Nakaharaku, Kawasaki 211, Japan

Recently, undoped semi-insulating GaAs crystals by LEC technique have attracted much attention for application in high speed IC and future optoelecronic IC. Many efforts have been directed towards the controlled and reproducible crystal growth technique of high quality GaAs.

We have successfuly developed a computer automatic diameter controlled (ADC) technique and a new in situ melt purification growth technique to obtain undoped semi-insulating GaAs single crystal with high reproducibility[1]. Highly homogeneous undoped semi-insulating GaAs single crystals were grown by computer ADC LEC technique linked with proposed new processes, which consist of an in situ melt purification and conductivity measurement of the melt for sensing the purification degree. This paper is concerned with the ADC LEC and the in situ purification growth and characterization of the undoped semi-insulating GaAs crystals. The growth technique and conditions for undoped semi-insulating crystal will be discussed and also reviewed.

In the new automated technique, growth parameters measured during growth were fed into the computer and processed in real time. The main responsibilities assumed by computer are, precise correction of weight signal and predicting control to cope with slow thermal response due to GaAs's low thermal conductivity. <100> GaAs single crystals of 2-inch in diameter could be grown in the MSR-6R type puller by closed-loop control using crystal weighing. Crystal diameter was regulated within ±1.5 percent (see Fig.1) even in the low temperature gradient furnace.

GaAs single crystals were grown from PBN or SiO_2 crucible in 3-50 atm using HB-grown polycrystal or Ga and As metals by ADC LEC technique. Impurity analysis of GaAs crystals revealed that GaAs melt in the crucible was purified by means of special distillation process in the chamber (see Table 1) and its degree of purification was detectable by the measurement of electrical conductivity of the melt. The resistivity of crystal pulled from the melt was also found to be varied with the measured melt conductivity as shown in Fig.2. Undoped semi-insulating crystals with resistivity 2-3x10^8 Ω-cm were reproducibly grown regardless of crucible materials and charged materials by this in situ technique.

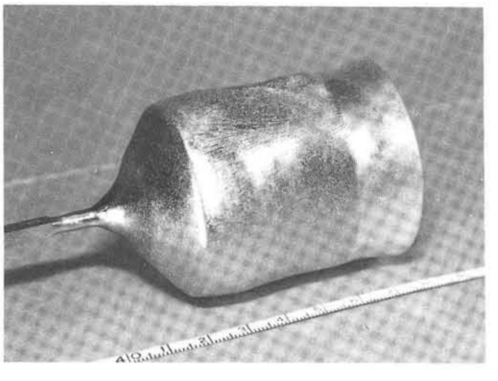

Fig.1. A typical <100> pulled GaAs single crystal by the ADC LEC technique

Table 1. Melt purification effect by the distillation.

Raw material	Crucible	B_2O_3 (water content)	Resistivity ($\Omega \cdot cm$)	Si Concn. ($atms \cdot cm^{-3}$)
GaAs Polycrystal	PBN	Dry	$< 10^2$	5×10^{15}
Ga, As	SiO_2	Dry	$< 10^2$	
			⬇ Distillation	
GaAs Polycrystal	PBN	Dry	$> 10^8$	(5×10^{14})
Ga, As	SiO_2	Dry	$> 10^8$	

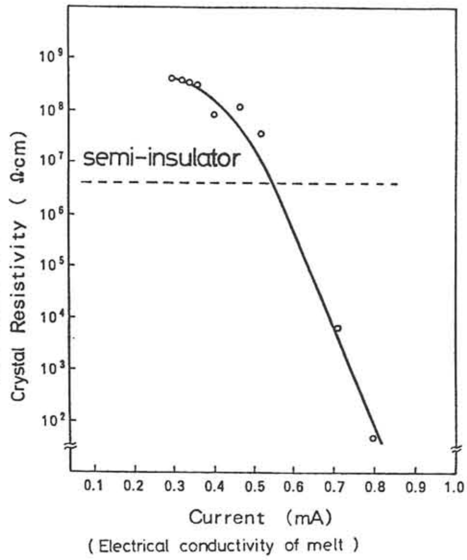

Fig.2. The relationship between GaAs crystal resistivity and electrical conductivity of GaAs melt.

Highly homogeneous undoped semi-insulating crystals from head to tail could be grown, while using PBN crucible, by minimizing As evaporation during growth both by using HB-grown polycrystal and by very low water content B_2O_3 ($\lesssim$100 ppm. H_2O). In Fig.3 the variations in electrical resistivity from head to tail part of the GaAs crystals are plotted against g (weight fraction solidified versus initial melt), as compared with that of commercially available one.

The crystals were characterized by etching and microscopic observation and photoluminescence measurement. Electrical properties were measured by van der Pauw's method. A correlation between vertical temperature gradient and dislocation density was investigated. The crystal grown in the low thermal gradient($\lesssim 70^{\circ}C/cm$), showed low dislocation density $1 \times 10^4/cm^2$. Detailed result of growth, electrical measurement and photoluminescence spectroscopy[2] of the crystal will be discussed.

References

1) K.Terashima, H.Nakajima and T.Fukuda; Proceedings of 2nd Conference on Semi-insulating III-V Material, 1982, to be published.
2) M.Tajima; unpublished.

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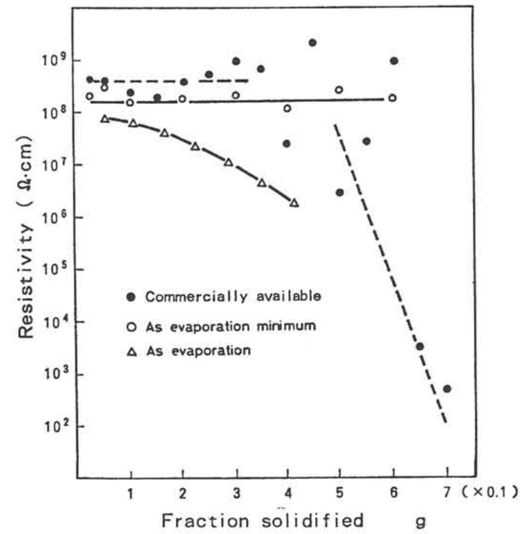

Fig.3. Resistivity of pulled GaAs crystals and commertially available crystal Vs g