GROWTH OF SEMI-INSULATING GaAs CRYSTALS WITH 100mm IN DIAMETER

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Semi-insulating GaAs single crystals of 100 mm diameter were pulled by HP-LEC and LP-LEC methods. Different growing conditions were investigated and the assessment of their influence on crystals parameters was done.

1. INTRODUCTION

Semi-Insulating (SI) gallium arsenide is widely applied in many semiconductor devices such as integrated circuits, metal semiconductor field effect transistors, laser optics elements *etc*.. The investigations are developed mainly in order to obtain the following features of crystals:

- large diameter: 4 inches [1] or even more [2] and increased crystal length [3,4],
- high structural quality, especially low dislocation density [5,6],
- good homogeneity of electrical and structural parameters [5,6].

Usually, SI-GaAs single crystals are grown by the Liquid Encapsulated Czochralski (LEC) method, but they may also be obtained using other methods eg. the Horizontal Bridgman (HB), the Vertical Bridgman (VB), the Horizontal Gradient Freeze (HGF) and the Vertical Gradient Freeze (VGF) [5,6,7].

Electrical parameters of SI-GaAs are determined by the balance between the concentration of deep EL2 donors, shallow donors and shallow acceptors (carbon) [7]. These parameters are also affected by the purity of raw materials (Ga, As), encapsulant (B_2O_3) and crucible material (quartz or pBN). The carbon concentration varies with the melt composition (Ga/As ratio) and the way of its preparation, as well as with the water content both in the boric oxide (B_2O_3) encapsulant and the ambient gas and the ambient gas pressure [8,9].

Structural parameters depend mainly on thermal conditions during growth. High dislocation density arises from thermal stresses generated during the growth process or due to dislocation loop formation resulting from precipitation of point defects,

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however thermoelastic stress is the dominant mechanism that gives rise to the observed dislocation density distribution [10,11,12]. Thermal gradients are mainly affected by the kind of ambient gas and its pressure, the geometry of thermal system (heater, crucible), crucible material and encapsulant layer thickness [3].

2. EXPERIMENT

The aim of this work was to elaborate the method for synthesis and monocrystallization of SI-GaAs crystals with diameter of 100 mm and to investigate the influence of some crystallization parameters such as the gas pressure and the crucible diameter on the structural properties of the crystals. The semi-insulating GaAs crystals of 100 mm in diameter were obtained using two ways:

- synthesis by the *injection* method and pulling by the Low-Pressure (LP) LEC technique,
- synthesis by the *in situ* method and pulling by the High-Pressure (HP) LEC technique.

2.1 HP LEC CRYSTAL GROWTH

The crystal growth processes were carried out in high pressure GALAXIE MARK-IV puller. The multiheater system contained three graphite heaters: main, lower and upper one (Fig. 1).

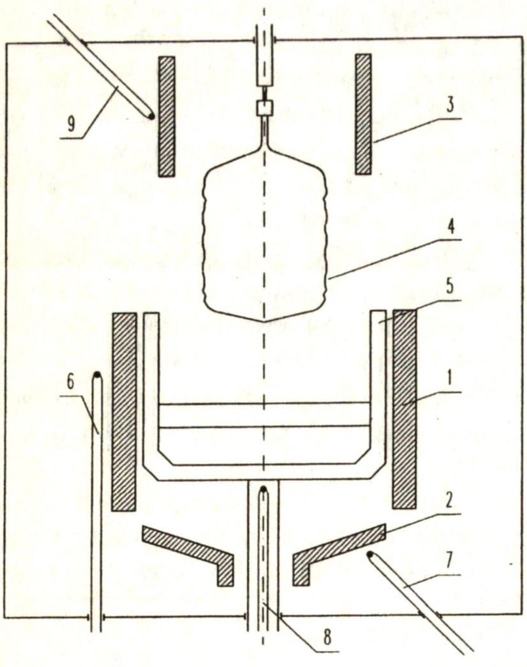

Fig. 1. HP-LEC multiheater system.
1- main heater,
2 - lower heater,
3 - upper heater,
4 - crystal,
5 - crucible,
6,7,8,9 - thermocouples.

All process parameters were programmed and controlled by the computer. The *in situ* synthesis parameters are shown in Figure 2.

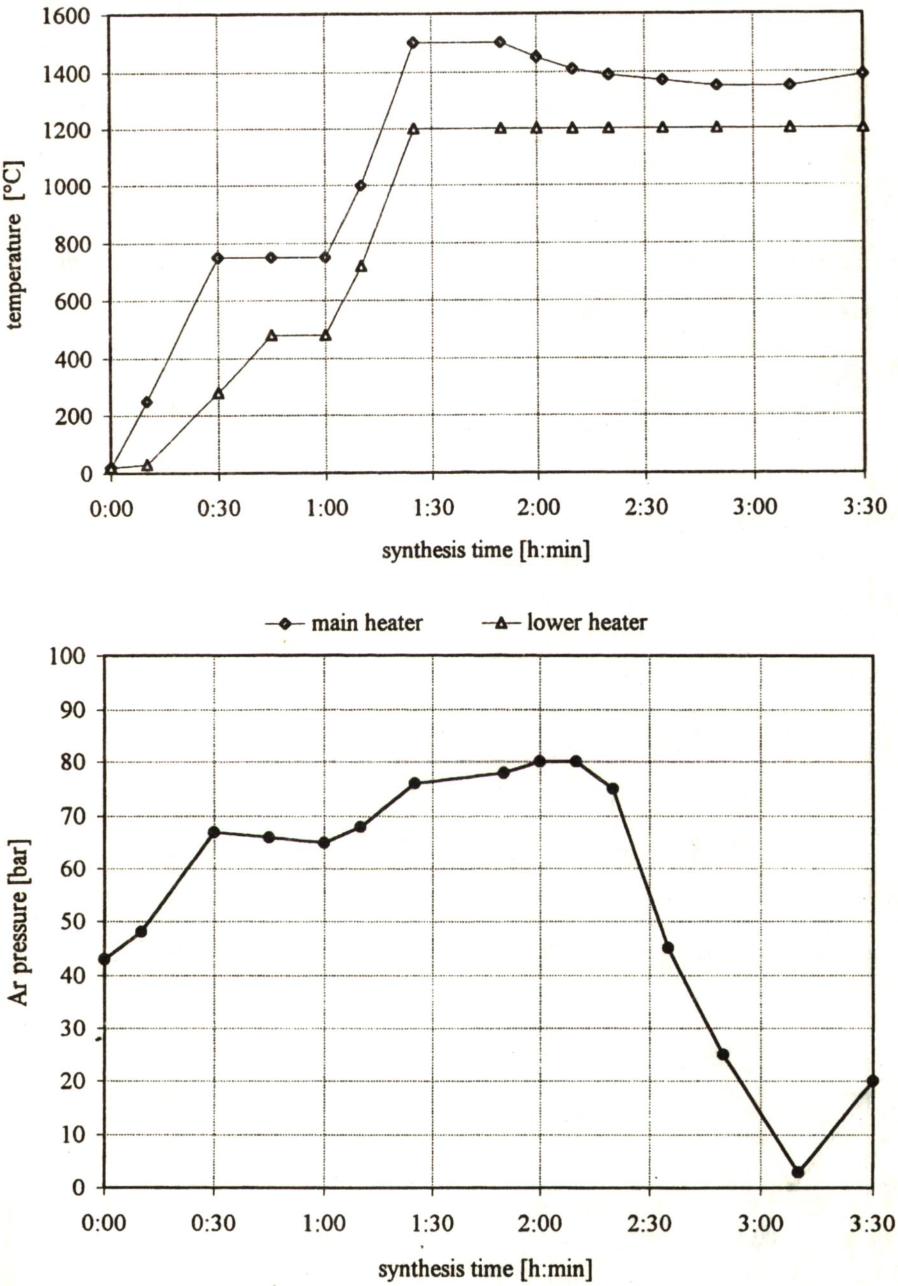

Fig. 2. Temperature and pressure changes during *in situ* synthesis.

Either the 6-in.-diam. or 8-in.-diam. quartz and pBN crucibles were used. The crystal growth parameters were as follows: charge weight 4900 g for 6" and 8000 g for 8" crucibles, 500 g B_2O_3 for 6" and 1000 g for 8" crucibles, seed orientation <100>, seed rotation 7 rpm, crucible rotation 15 rpm, pulling rate 8 mm/h and Ar pressure 15 bar.

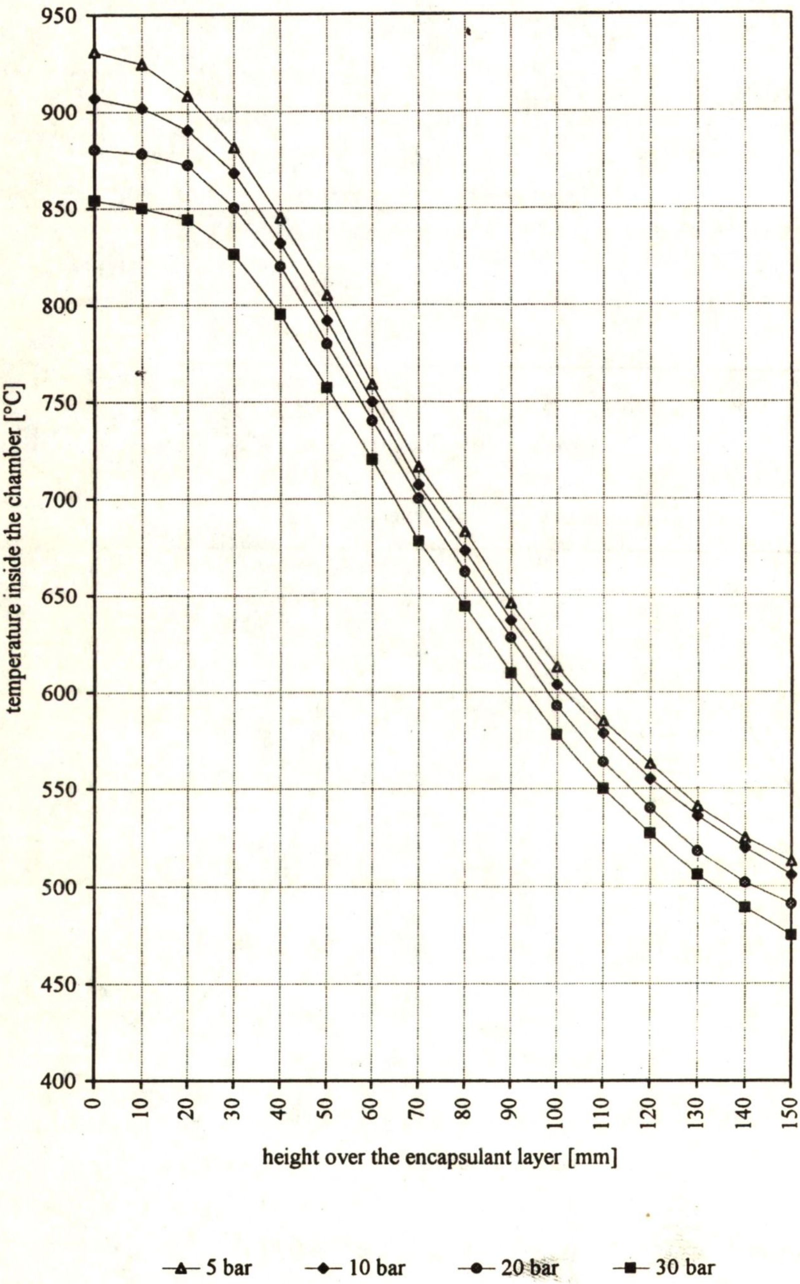

Fig. 3. The influence of Ar pressure on the axial temperature profile over the encapsulant.

Temperature profiles measured for some Ar pressures inside the chamber are shown in Figure 3. The measurements were made in seeding conditions (*i.e.* the temperature of molten GaAs surface 1240°C). It can be seen from figure 3 that the temperature gradients inside the encapsulant layer increase with the inert gas pressure. For Ar pressure of 5 bar they are 155°C/cm, for 10 bar 165°C/cm, for 20 bar 190°C/cm and for 30 bar 195°C/cm. It is very important, with the increase of thermal gradients, because along thermal stresses are generated at the crystallization front and in this way the quality of the crystals is getting more imperfect [10].

2.2 LP LEC CRYSTAL GROWTH

The crystal growth processes were carried out using low-pressure GK-2 pullers equipped with only one graphite heater (Fig. 4).

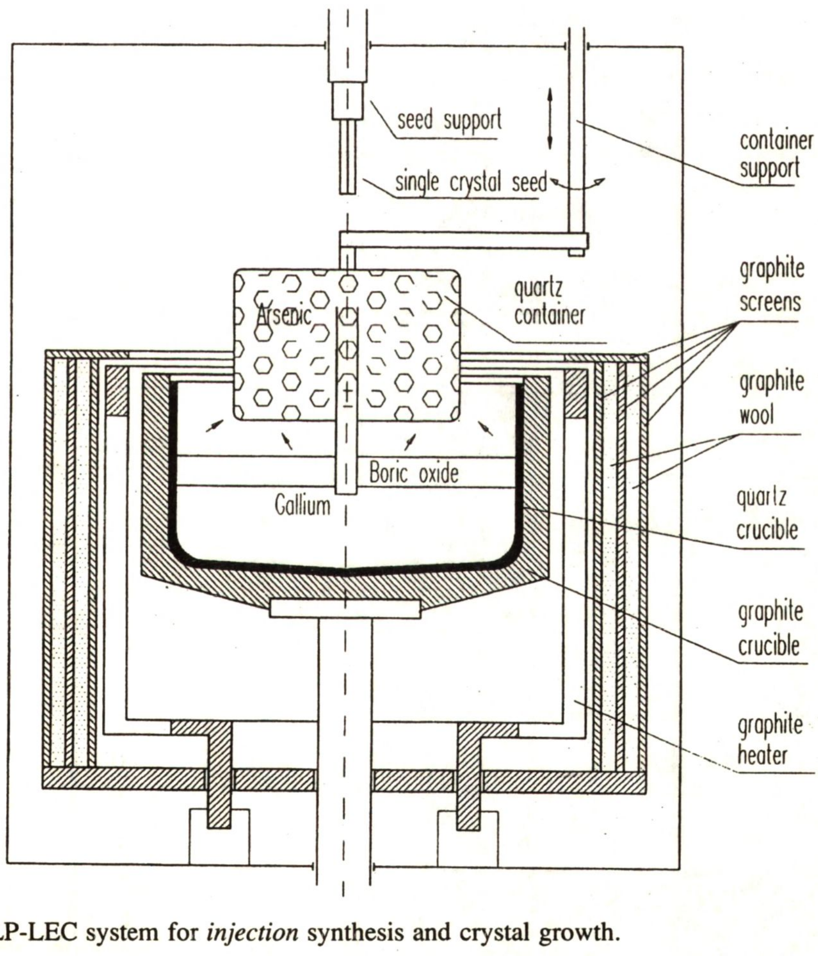

Fig. 4. LP-LEC system for *injection* synthesis and crystal growth.

The quartz and pBN crucibles of 6" in diameter were applied. The crystallization processes parameters were as follows: input charge 4300 g, encapsulant 450 g, orientation <100>, seed rotation 12 rpm, crucible rotation 1.5 rpm, pulling rate 12 mm/h, Ar pressure 1.3 bar.

3. RESULTS AND DISCUSSION

The single crystals of about 4 kg and 7.5 kg (from 6" and 8" crucibles respectively) were obtained. The “as grown” crystals were then annealed and their parameters are shown in table 1.

Table 1. Typical parameters of the SI-GaAs crystals.

Crystal		crucible diam. [in]	ρ [Ωcm]	μ [cm^2/Vs]	$N_c \times 10^{15}$ [cm^{-3}]	E P D [cm^{-2}]
1754	I	6	2.28×10^7	6060	-	3.5×10^4
LP	II					9.7×10^4
1822	I	6	1.91×10^7	5460	2.5	4.4×10^4
LP	II		1.26×10^6	3520	2.1	8.8×10^4
1829	I	6	8.63×10^7	6770	0.7	3.0×10^4
LP	II		8.32×10^7	6160	7.9	9.1×10^4
1834	I	6	7.41×10^7	4200	8.1	2.1×10^4
LP	II		9.50×10^7	5140	16.0	7.1×10^4
1865	I	6	4.06×10^7	5150	1.4	4.2×10^4
LP	II		3.36×10^7	5360	3.5	9.5×10^4
1805	I	6	3.42×10^6	6090	1.3	4.6×10^4
HP	II		5.35×10^6	4170	1.5	1.3×10^5
1808	I	6	1.58×10^8	5360	5.2	5.3×10^4
HP	II		7.73×10^7	6170	3.5	1.8×10^5
1823	I	8	5.04×10^7	5130	9.0	1.8×10^4
HP	II		3.53×10^7	4330	20.0	5.9×10^4
1874	I	8	1.82×10^8	4280	5.4	4.4×10^4
HP	II		1.28×10^8	4320	13.0	8.3×10^4
1875	I	8	1.75×10^8	3720	20.9	2.6×10^4
HP	II		8.67×10^7	3530	16.0	6.8×10^4
1878	I	8	4.23×10^7	4150	1.2	4.5×10^4
HP	II		2.0×10^{-1}	3970	2.0	6.9×10^4

The crystals obtained in similar conditions in different pullers have similar properties.

The influence of ambient gas pressure and the crystal to crucible diameter ratio on temperature gradients and in the same way on the crystal structural quality has been observed.

The comparison was also made between the structural quality of the crystals grown

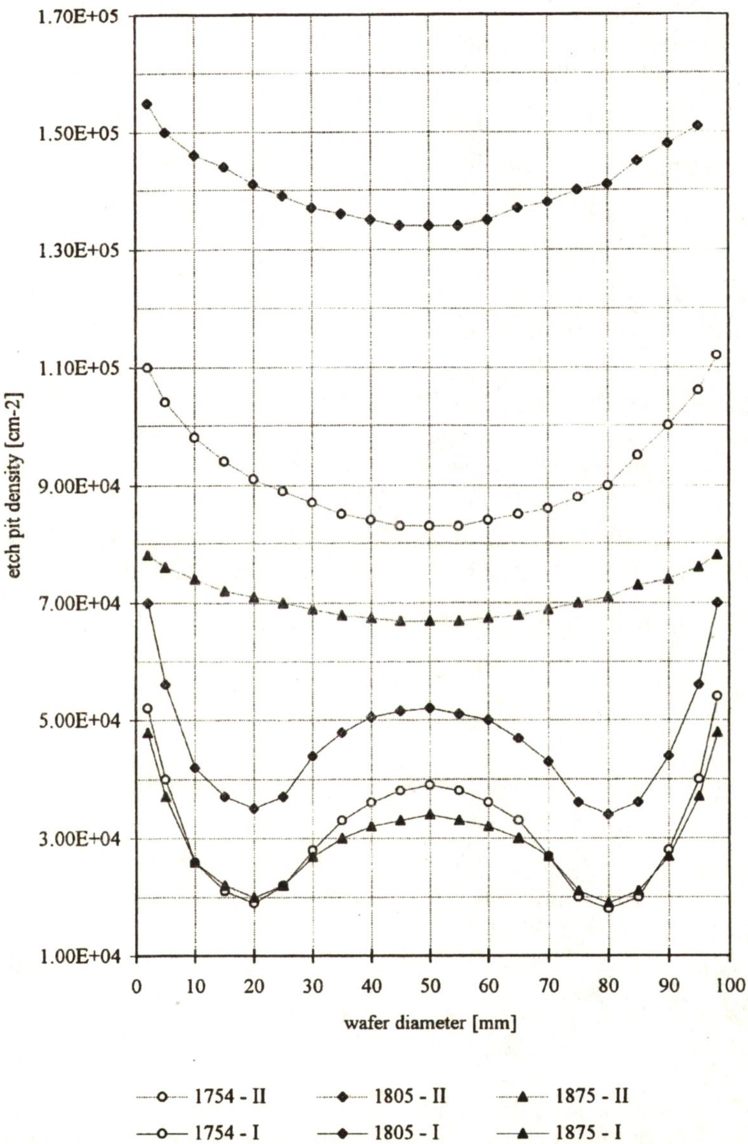

Fig. 5. Typical EPD profiles for 4" crystals grown in different conditions.

by HP-LEC and LP-LEC techniques in the similar heating systems and from the crucibles of the same diameter. It was found that the crystals grown by the high-pressure technique exhibit higher dislocation density (see Fig. 5: "1805" HP-LEC and "1754" LP-LEC). This is due to the thermal stresses.

The structural quality is also influenced by the crystal to crucible diameter ratio. The crystals of the diameter over 4" pulled from the crucibles of 6" in diameter are of worse structure quality in comparison with the similar crystals grown from the bigger, 8" in diameter crucibles (see Fig. 5: "1805" - 6" crucible and "1875" - 8" crucible).

The above statements are confirmed by the results of Etch Pit Density (EPD) measurements shown in Table 1. Typical EPD profiles are presented in Fig. 5.

4. CONCLUSIONS

The process parameters have been established for the GaAs synthesis by the low-pressure *injection* method (for 5 kg of charge) and the high-pressure *"in situ"* method (for 5 kg and 8 kg of charge).

The influence of some factors such as the ambient gas pressure and the ratio at the crystal diameter to crucible diameter on the crystal structural perfection was determined.

The physical properties of the obtained single crystals were comparable with those given by the other producers.

The aim of our future research will be as establishing the crystal growth conditions to obtain single crystals of more than 4" in diameter and lowering the dislocation density.

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SUMMARY

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Semi-insulating GaAs single crystals of 100 mm diameter were pulled by HP-LEC and LP-LEC methods. Different growing conditions were investigated and the assessment of their influence on the crystals parameters was done.

СОДЕРЖАНИЕ

ВЫРАЩИВАНИЕ ПОЛУПРОВОДНИКОВЫХ КРИСТАЛЛОВ GaAs ДИАМЕТРОМ 100 мм

Полупровадниковые монокристаллы арсенида галлия диаметром 100 мм получены методом Чохральского при низком (HP - LEC) и высоком (HP - LEC) давлениях инертного газа в камере.

В работе представлены влияния некоторых параметров процесса (диаметр тигля, давление в камере) на свойства полученных монокристаллов.