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The latest development in the preparation of indium phosphide (InP) polycrystals and single crystals

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Abstract InP crystal is an increasingly important semiconductor material in the application of long-wave optoelectronic and high frequency devices. The equilibrium vapor pressure of phosphorus at the melting point of InP is so high that the synthesis process is very difficult. Liquid-encapsulated Czochralski (LEC) pulling from the melt at high pressure is a generally favored technique to grow InP single crystals. This technique involves two steps: the synthesis of polycrystalline powder and the growth of single crystal from the melt at high pressure. This article reviewed the latest development in the preparation of InP crystal and the evaluation on the crystal quality.

Key words InP, Liquid-ecapsulated Czochralski (LEC), Synthesis, Evaluation

1. Introduction

Indium phosphide (InP) was developed later than GaAs and GaP. Compared with other III-V compounds, InP has some superior physical properties, such as suitable lattice constants and band gap, large electron drift velocity, large thermal conductivity, and so on (Table 1). Because of these characteristics, InP is now used in industrially for laser diodes (LDs), light emitting diodes (LEDs), avalanche photodiodes (APDs), P-I-N detectors as well as promising radiation-resistant solar cells [1]. The development of these devices requires substrate with high crystal quality and with large diameters of more than 3 inches for low cost device fabrication. In addition, long-length single crystals are also required from the viewpoint of the substrate manufacturing cost.

The liquid encapsulated Czochralski (LEC) method was first used to grow InP semiconductor single crystals by Mullin in 1968 [2]. Although this method was more costly than zone melting or gradient freeze, it had the distinct advantage that oriented crystals could be grown in a single working day. Single crystals were obtained at a much higher yield when stoichiometric polycrystalline starting materials were used. The common defects in InP crystals are twin, dislocation and some point defects related with nonstoichiometry. The main problems and corresponding measures in the preparation of InP poly and single crystals are summarized.

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2. Synthesis of InP Polycrystals

2.1. The requirement for InP polycrystals

In the phase diagram of In-P binary system [3], there is only one compound, InP, its melting point is 1062°C (Fig. 1). The melting point of pure In is 156°C, but no melting point exists for phosphorus at 1 atm. due to its sublimation. Phosphorus has many allotropes, such as red, white, and black phosphorus. Red phosphorus is the most widely used allotrope for the synthesis of InP owing to safety considerations. Red phosphorus sublimes and becomes a gas when it is heated over 416°C. The dissociation pressure of phosphorus is as high as 25~27.5 atm at the melting point.

Because of this higher dissociation pressure, it is difficult to synthesize indium and phosphorus directly in the high-pressure crystal puller. Therefore, high purity indium and high purity phosphorus are synthesized in a high-pressure chamber to InP polycrystal raw material and then melted in a high-pressure puller to grow single crystals (i.e. two-step method). In order to grow high quality InP single crystal, following requirements must be satisfied:

1) Purity of polycrystal should be as high as possible. From the viewpoint of industry, purity, measured as car-

Table 1
Physical properties of InP crystals [1]

Crystal structure	Zinc blende	Band gap (eV) at R.T.	1.35
Lattice constant (A)	5.869	Optical transition	direct
Density (g/cm^3)	4.8	Specific dielectric constant	12.5
Melting point (°C)	1062	Heat capacity (cal/K)	0.073
Vapor pressure (atm) at M.P.	27	Intrinsic resistivity ($\Omega \cdot$ cm)	8×10^7
Linear expansion coefficient (/°C)	4.5×10^{-6}	Hole mobility (cm^2/V · s)	150
Thermal conductivity (W/cm · °C)	0.70	Electron mobility (cm^2/V · s)	4500
Temperature dependence of band gap (eV/°C)	-2.9×10^{-4}	Intrinsic carrier concentration at R.T. (cm^{-3})	8×10^7

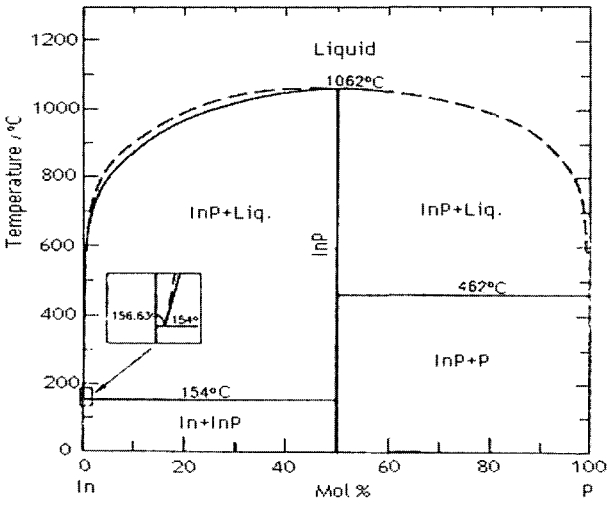

Fig. 1. Phase diagram of In-P binary system [3].

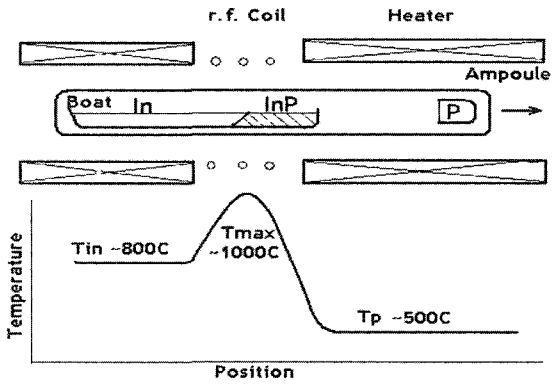

Fig. 2. The synthesis of InP polycrystals with Horizontal Bridgman method.

rier concentration, must be maintained at least less than 10^{16} cm^{-3}, preferably, less than 5×10^{15} cm^{-3}.

2) Indium inclusions, that is, un-reacted indium, have to be as few as possible; preferably they should not be observed by optical microscopes.

3) The batch quantity should be as large as possible, with high synthesis rate from the viewpoint of production cost.

2.2. *Horizontal Bridgman (HB) technique*

The furnace has three zones: indium melting zone, synthetic zone and phosphorus vapor control zone. High-purity indium in a quartz boat or a pBN boat and the high-purity phosphorus ingot are sealed in a quartz ampoule and heated in the synthesizer. Each zone is heated up, for example, to the temperature indicated in the Fig. 2. During heating, the vapor pressure of phosphorus is raised higher than the atmospheric pressure. Therefore, inert gas such as N_2 or Ar should be introduced during the temperature increase in the synthesizer in such a way that the high vapor pressure of phosphorus in the ampoule can be balanced to prevent the rupturing of the ampoule.

In HB technique, to obtain InP polycrystals of reasonable purity, possessing few indium inclusions, and having a reasonable synthetic rate, the following parameters must be optimized:

1) indium melt temperature
2) phosphorus vapor pressure
3) ampoule transfer rate
4) synthesis batch quantity
5) boat material

HB technique is a very high growth rate synthesis method. However, since this method requires a temperature higher than the melting point, the purity is usually degraded due to contamination by silicon from the quartz boats. The contamination by silicon will result in the In_2O_3 particles:

$$4In\ (melt) + 3SiO_2 = 3Si\ (in\ In\ melt) + 2\ In_2O_3 \quad (1)$$
$$4In\ (gas) + 3SiO_2 = 6SiO\ (gas) + 2\ In_2O_3 \quad (2)$$
$$3SiO + 2In\ (melt) = 3Si\ (in\ In\ melt) + In_2O_3 \quad (3)$$

However, if the quartz boat is replaced by pBN and the synthetic conditions are well optimized, it is possible to grow high-purity InP polycrystals. So, HB technique is now industrially accepted.

In addition, this method can also be used to grow InP

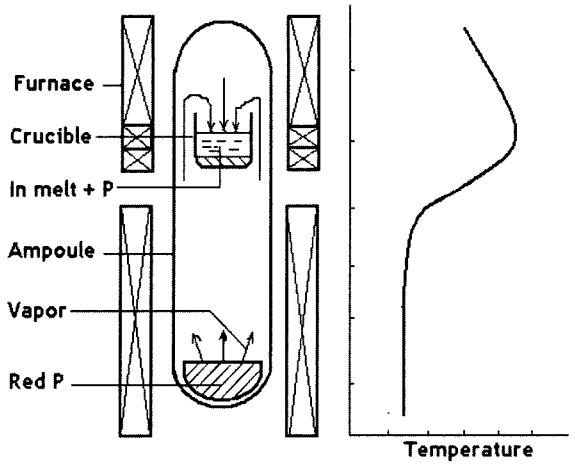

Fig. 3. Principle of synthesis of InP polycrystals by the solute diffusion technique [1].

single crystal [4], however, the thermal stress from the difference of the thermal expansion coefficient between InP and quartz crucible can generate high dislocation density of 10^5~10^6 cm^{-2} [5, 6].

2.3. Synthesis by Solute Diffusion (SSD) technique

Indium and phosphorus are sealed under vacuum in a quartz ampoule, the phosphorus is heated at a certain temperature in order to obtain the phosphorus vapor with pressure less than one atmosphere. The phosphorus is resolved in the indium melt to the saturation composition. Since the temperature at the bottom of the crucible is lower than that at the surface of the melt, dissolved phosphorus is diffused from the melt surface to the bottom of the crucible. When the composition of phosphorus at the bottom of the crucible is increased, and it exceeds the saturation composition, InP polycrystals are grown from the bottom of the crucible (Fig. 3).

SSD technique has an advantage that the growth can be performed at lower temperature and the contamination by silicon from the quarts is less, so it is possible to obtain ultra-pure InP polycrystals. However, since the diffusion coefficient of phosphorus in the indium melt is very small, the growth rate of this technique is very slow. This is why SSD technique is only applicable at laboratory, and is far from commercial production.

3. Growth of InP Single Crystal

Liquid-encapsulated Czochralski (LEC) has been widely used for the production of InP semiconductor crystals.

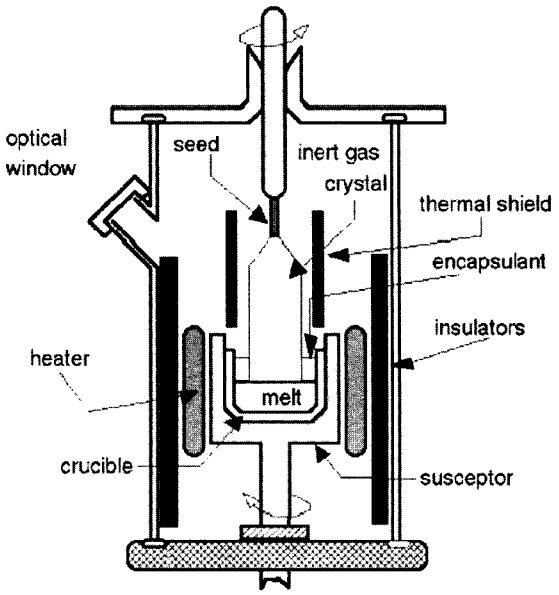

Fig. 4. Typical cross view of the LEC technique.

In LEC technique (Fig. 4), B_2O_3 glass is used as an encapsulant to prevent the loss of phosphorus from the melt. Single crystals are grown from the InP melt covered with a liquid B_2O_3 layer and under the high pressure of inert gas. The diameter of the crystal is calculated using the weight of the growing crystal, which is measured by a weight sensor equipped on the pull rod. The shape of crystals can be controlled by adjusting both the temperature at the growth interface and the pulling rate of the crystal.

3.1. Process of LEC method

1) Treatment of polycrystal: Since the polycrystals used as source materials are usually covered with oxidation layers, they must be treated with etchants and cleaned just before being loaded into the crucible. Then, the treated polycrystals must be kept in an inert atmosphere.

2) Setting up the hot zone: The Cz furnace is built in such a manner as to establish a temperature profile having cylindrical symmetry, it is necessary to check any slight deviations of the hot zone from the standard alignment and whether or not the center of rotation of the seed crystal coincides with that of the crucible.

3) Doping: Since sulphur has a large effect on reducing the EPD, sulphur-doped crystals are mainly used for photodetectors. To obtain whole area dislocation-free crystals, sulphur concentration must be made larger. However, if the doping concentration is increased, the grown crystals are easy to twin so there is a maximum

limit for the doping. In the [100] direction, the doping limit is 5×10^{18} cm^{-3}, and in the [111] direction, the limit is 3×10^{19}. So, in [111] direction, it is easier to obtain a large dislocation-free area.

4) Loading: The etched polycrystals and dopants are loaded into the bottom of a quartz or pBN crucible and are then covered with an encapsulant of B_2O_3. Particular attention must be paid to the water content and temperature of the B_2O_3.

5) Attaching of seed: InP seed crystal, after being etched in aqua region, is attached to the seed holder. The seed crystal usually has a [001], [111] or [110] direction.

6) Evacuation of chamber: The chamber is closed and evacuated down to 10^{-2}~10^{-3} torr.

7) Heating: The crucible is heated, under high pressure (30~50 atm.) of inert gas (such as N_2 and Ar), to melt the B_2O_3 encapsulant and the source materials. The temperature of InP melt is stabilized at near seeding temperature, whereupon the seed crystal is brought in to contact with the surface of the melt.

8) Diameter and shape control: Diameter and shape control of the growing system is automatically performed by a computer system. Several sensors are equipped to measure the growth parameters, such as temperature, pressure, crystal weight, pulling rate and rotation rate of both the crystal and the crucible. These parameters are put into computer, treated in real time, and the signals are then fed back to both the heating and control systems.

9) Cooling: The cooling rate of the furnace after growth must be carefully determined, since rapid cooling increases the dislocation density in the crystal, even though rapid cooling is essential for production.

The advantage of LEC method is that single crystals with large diameter can be grown with high growth yield, so far, 4 inch diameter crystals are mainly produced in industry. However, this method is difficult to grow crystals with low dislocation density, because of its high radial and axial temperature gradient near the liquid-solid interface. In order to obtain InP crystal with low dislocation density, vertical gradient freezing (VGF) [2] and phosphorus vapor controlled LEC (PC-LEC) [7] were invented in recent years.

In practice, the source polycrystals used for LEC growth are produced by HB method. This process is commonly called "two-step" technique. This technique is very expensive. Moreover, the contamination by impurity is inevitable. So recently, one-step synthesis and growth technique, i.e., pulling a single crystal from an in situ synthesized InP melt, has been widely investigated by some researchers [8].

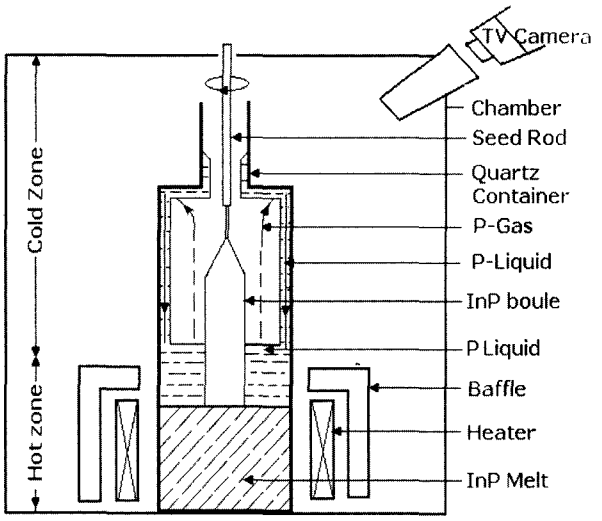

Fig. 5. Schematic diagram of liquid phosphorus encapsulated Czochralski method.

Figure 5 is a liquid phosphorus encapsulated Czochralski (LP-Cz) method invented by Tomoki and Tsuguo [1]. The starting materials are solid red phosphorus and solid indium. When heated, the indium melts first and then the phosphorus sublime, generating phosphorus gas. When the gas reaches to the inner wall cooled by cycling water, it condenses into a liquid and flows down on to the indium melt. The liquid phosphorus reacts with the indium melt to produce indium phosphide. Here, the liquid phosphorus layer is used as an encapsulant to prevent phosphorus evaporation from the melt.

3.2. Suppression of twinning

InP single crystals have a zincblende structure as shown in Fig. 6 [9]. In this structure, P atoms arrange as face cubic closed packing, and In ions occupy the 1/4 tetrahedral interstitial positions. The four nearest-neighbor bonds are equal, and each pair of bonds meet at a bond angle of 109°. In-P bond possesses not only covalent bonding, but also ionic bonding. The cleavage plane of InP is {110}, because it has electrical neutrality. Since each double layer of the stacking sequence consists of one sheet of In atoms and one sheet of P atoms, InP structure is characteristic of face polarity, that is, In plane and P plane.

Twinning in the zinc blende semiconductor lattice is represented a 60° rotation about the normal to the {111} plane. The generation of twinning is the most troublesome problem in growing InP single crystals. This is

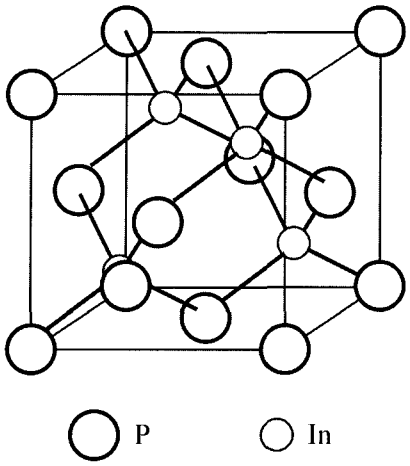

Fig. 6. Crystal structure of InP.

because InP has a very small stacking fault energy (18 mJ/m^2) [10] and so twin boundary is easily formed during the crystal growth, especially when [100] oriented single crystals are grown. Hurle proposed that these twins nucleate in regions where {111} edge facets are anchored to the three-phase boundary (TPB) and that twinning produces a {111} oriented surface on the region of the crystal [11]. Although the mechanism of twinning has not been clarified yet, it is empirically noted that following conditions relate to the occurrence of twin [12].

1) B_2O_3 purity: B_2O_3 with extremely small amount of water is recommended for the twinning prevention.

2) Melt composition stoichiometry: In the case of InP, the melt composition is substantially changed toward the indiumrich side, if this deviation is too large, the twinning and/or polycrystallization takes place easily because of supercooling. So, it is necessary to use InP polycrystals of a stoichiometric composition as source materials.

3) Temperature distribution: For growing twin-free single crystal, a larger axial temperature gradient is desired. However, it is undesirable for decreasing dislocation density. On the other hand, radial temperature distribution affects the shape of solid-liquid interface, when it is small, the interface shape becomes flatter, and when it is larger, the interface shape becomes convex toward the bottom of the crucible. It is suggested to keep the shape of the convex to the melt for prevention of twining [13].

4) Temperature fluctuation: Temperature fluctuation is also a factor to affects the occurrence of twinning. It is very difficult to minimize the temperature fluctuation, but this must be achieved for the prevention of twinning.

5) Growth rate: Decreasing the growth rate is a way to prevent twinning, but this is not recommended since it also decreases the yield of crystals.

3.3. Reduction of dislocation density

The presence of dislocations in semiconductors produces dangling bonds, which affect the carrier concentration and mobility in semiconductors. Therefore, dislocations in InP deteriorate performance and stability, and reduce the reliability and lifetime of semiconductor devices [14]. Dislocation generation in LEC-grown crystals can result from different sources, which include excessive thermal stress, native defects, nonstoichiometry, dislocation propagation from highly dislocated seed and larger diameters. Sometimes, a casual relationship exists between twins and dislocations [15]. However, the effect of nonstoichiometry and native defects upon dislocation generation has not yet been demonstrated for LEC-grown InP. The dislocation propagation from the seed can be eliminated by using dislocation-free seeds and the so-called "**necking**" technique. Only the thermal stress is the main cause of dislocation.

During LEC growth, the crystal is subjected to "thermally induced stress" due to the large thermal gradient near the solid-liquid interface. The excessively cooled crystal periphery produces thermal contraction, and, consequently, the periphery is in tension, but the hot core in compression. As soon as this thermal stress in the {111}, [110] primary slip system exceeds a critical value, **the critical resolved shear stress (CRSS)**, the crystal experiences plastic flow, and the excessive thermal stress are released by crystallographic glide.

Consequently, there are two possible routes to decrease the dislocation density in InP crystals by (1) decreasing the thermal gradient, and, consequently, the thermal stress inside the crystal to a value lower than the CRSS, and (2) increasing the CRSS via a lattice hardening, or "impurity-hardening", mechanism.

1) Lowering the thermal gradients: The low thermal gradient can be achieved by the following ways,

- Increasing the boric oxide quantity
- Using multi-heater furnace
- Using thermal shield
- Control the gas nature and its pressure
- Adjust the crucible and crystal rotation rate

2) Doping to suppress the dislocation: Impurity-hardening to increase the CRSS can be achieved by doping some elements. This effect was explained to be **pinning effect**. That is, when the energy between a substituted impurity atom and a host atom exceeds the bond

energy between two host atoms at the lattice site, the dislocation are bound by impurity atoms and become relatively immobile. So, doping can suppress the occurrence and the propagation of the dislocation in crystals.

The most effective dopant was found to be Zn, followed by S and Te. In optical device usage, Sn or S is added for n-type substrates, and Zn is added for p-type substrates [16]. It is noteworthy that the segregation coefficient and lattice parameter variation must be considered in the selection of dopants. For example, the minimum sulfur doping concentration necessary to get rid of dislocations is 5×10^{18} cm^{-1}, however, due to the low segregation of sulphur in InP, 0.5, its concentration at bottom will reach 5×10^{19} cm^{-1}, such high doping level will not only induce optical absorption at the wavelength of 1.3 μm, but also induce strains in the epilayer because of the lattice parameter variation respected to the undoped materials.

4. Evaluation on InP crystal quality

4.1. EPD measurement

Etch-Pit Density (EPD) is one of the best indicators of the quality of III-V semiconductor crystals. In this measurement, a wafer is chemically etched to reveal etch pits, which are then counted under an optical microscope. In addition to the etch pits, other defects revealed on the etched surface, such as inclusion, precipitation and micro-twin must be carefully observed. EPD measurement process is briefly outlined as follows:

1) Wafers, cut perpendicularly to the direction of growth, are polished.

2) The polished wafers are etched with an **H_3PO_4-HBr** aqueous solution at room temperature for a few minutes.

3) The number of etch pits on a wafer is carefully counted under an optical microscope. Usually, this counting process is carried out at many points on the wafer in order to learn the EPD distribution and to evaluate its uniformity.

4) The number of etch pits is converted into an EPD (number of etch pits per 1 cm^2) value.

5) From the results, the single crystals are classified into various grades.

4.2. Hall Effect measurement

This measurement can be used to determine the conduction type and classify the fundamental quantities of semiconductors, namely, electric resistivity, carrier concentration, and Hall mobility. From these results, we can confirm how much residual impurity is included in the undoped crystal and whether or not the crystal is properly doped. Generally, the Hall effect measurement is carried out by measuring the voltage induced when mutually perpendicular electrical and magnetic fields are applied to the samples. The *Van Der Pauw* method is widely used because it can accommodate flat samples of any shape.

1) Pretreatment: Wafers are polished and etched.

2) Preparation of samples: The wafer is cut into square chips (4×4 mm^2). For n-type InP, Au-Ge-Ni alloy (in the form of electrodes) is evaporated at the chips four corners. For p-type InP, Au-Zn alloy is used. After this, the chips are annealed in an atmosphere of nitrogen for 20 minutes. The annealing temperature is 450°C.

3) Measurement: Each of the chips electrodes is connected electrically to an instrument.

4.3. Crystallographic characterization

Evaluation of crystal perfection is essential since the properties of devices strongly dependent on the structural perfection of the single crystal. Detailed information is obtained by using X-ray or electron microscopy techniques. The X-ray topography technique reveals imperfections, such as dislocations, subgrain-boundary, striations and lattice strains [4].

The x-ray double crystal rocking curve method is useful in that it gives quantitative measure of crystal perfection. However, the surface of crystal must be carefully treated, since this method only provides information for the areas, usually on the surface, that is affected by the X-ray beam. This, in turn, depends on the beams penetration depth.

Recently, synchrotron radiation X-ray white beam has been widely used in material characterization due to its high intensity, wavelength tenability and fine geometric resolution [17]. The lattice constants of single crystals have been precisely determined by the X-ray diffraction method, which provides information concerning crystal stoichiomery.

4.4. Characterization of impurities

Impurities seriously affect the characterization of semiconductor materials and their devices. Therefore, it is essential to identify the kinds of impurities present and

to determine their amount. Many kinds of instrumental analysis are applied to these purposes.

1) Mass spectroscopy

Spark source mass spectroscopy (SSMS): sensitivity 0.01 to 0.1 ppm.

Second ion mass spectroscopy (SIMS):

Glow discharge mass spectroscopy (GDMS):

2) Atomic absorption/atomic emission spectroscopy

Graphite furnace atomic absorption spectroscopy (GFA)

Inductively coupled plasma atomic emission spectroscopy (ICP)

4.5. Optical evaluation

Optical measurements are a nondestructive method and generally have high sensitivity and high spatial resolution. Optical properties of semiconductors are strongly related to the band structure, which includes band gap, and energy levels formed by impurities. Therefore, optical measurements are useful tools for understanding electrical properties. They also provide information about defects in a single crystal. It is essential to fully understand the optical properties of InP single crystals, since they are widely applied in various optical devices.

1) Photoluminescence (PL) technique: PL is emitted when excess electrons and excess holes are recombined after their separation due to the crystals surface being exposed to light whose energy is greater than the band gap energy. The PL measurements are often employed to identify impurities in crystal as well as information concerning defects, strains and their position uniformities in a crystal. However, this technique has two drawbacks: one is difficulties in analyzing the result quantitatively and the other is useless in the detection of non-radioactive impurity centers.

2) Transmittance measurement: When InP single crystals are utilized for optical devices, high transmittance through a substrate is necessary. Optical transmittance in the infrared region depends on the free carrier concentration.

3) Infrared light scattering measurements: This technique can be used to observe the small-size inclusions, aggregations of defects, and precipitates.

4.6. Computer simulation

With the development of high speed, high capacity supercomputers, more and more crystal growers can simulate both the temperature profile of their furnaces and the growth conditions [18-20]. On the basis of the predication of their simulations, they can employ an appropriate hot zone structure and the most suitable growth conditions. And consequently, they can obtain high quality single crystals from the first growth attempt, without repeatedly trying troublesome growth experiments. However, computer simulation cannot strictly reproduce such growth situations because there are many unknown factors and insufficient physical properties. But, without using the complex three-dimensional analysis, useful information can still be obtained through a simple, one or two dimensional heat flow analysis [21].

5. Conclusion

InP is an important semiconductor crystal in the application of optical devices and high-speed circuits. It can be produced with LEC method in industry. The main defects existed in InP crystals are twins and dislocations. Even though their forming mechanisms have been unclear so far, the crystal quality can be improved by optimizing the growth conditions.

Acknowledgements

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