Detached Growth: Unfolding Four Decades Growth Mystery into Vertical Directional Solidification Technique on Earth

D. B. Gadkari

Department of Physics Mithibai College Mumbai-400056 India

Abstract- Since 1993, the vertical directional solidification (VDS) technique has been shown experimental evidences for the total detached growth for InSb/GaSb ingots grown- without seed, without wall contact, without coating and without external pressure. Detached growth and apparition of spontaneous gap performs a trick to the entire ingot by the self-detached growth and self-pressure difference. Among the ingots grown by VDS technique, 80% ingots slide out easily, 15% ingots were entrapped in the conical region of the ampoule, and 5% ingots were attached to the ampoule wall. Three types of detached growths have been investigated. Concepts of meniscus conversion from concave to convex and concave crystal-melt interface shape have been predicted from the strong evidence of experimental analysis. Experimental statistics for the detached growth and its mystery of the four decades is unfolded. The qualitative physical model has been proposed on the basis of experimental statistics as "A new crystal growth process"

Index Terms- A1. Electronic materials, A1. Semiconductors, B1. Crystal growth, B1. Solidification, C1. Etching, C1. Optical microscopy, D1. Crystal structure, D1. Hall Effect

I. INTRODUCTION

1.1 Crystal growth in space

The vision and mission of manufacturing perfect crystals in space began in the 1960's in the Apollo era and manufacturing of semiconductor materials continued in the 1970's. On the earth, a major problem in semiconductor device manufacturing was variation in impurity doping on both macroscopic and microscopic scales. Directional solidification in microgravity has repeatedly showed detachment of solids that grew without complete contact with the ampoule wall. In occurrence of detachment, dislocation densities were greatly reduced, and the nucleation of new grains and twins were sometimes completely eliminated. In 2001, reports were published on "Microgravity effects on materials processing: A review" by Wilcox and Regel. A comprehensive survey of the detached crystal growth in microgravity is cited in literature [1].

1.2 Detached crystal growth

The detached growth was first observed spontaneously in space experiments performed on NASA Skylab-III mission-1974 [2]. It was established that diffusion-controlled, steady state segregation was achieved during InSb and InSb:Te solidification, which never accomplished on earth. A mechanism involving the semiconductor growth angle, its wetting angle at the crucible wall and the crucible surface roughness is reported for de-wetting of InSb in microgravity by Duffar et.al. [3]. Regel and Wilcox [4] had proposed the Moving Meniscus Model (MMM) to explain the detached phenomenon. In MMM, there is a gap between the ingot and the ampoule wall, but the melt remains in contact with the ampoule wall [5]. The pressure difference is developed by entry of dissolved gas into the gap, which is rejected by the growing solid, and released across the meniscus. Duffar et.al. [6] performed solidification of InSb, GaSb and GaInSb alloys under microgravity condition to study the chemical segregation. On the basis of experimental result, semiconductor could have high contact angles due to slight pollution (Duffer) of gases. Another phenomenon based on pressure difference between the hot and cold sides of melt (Wilcox) were proposed for the thermodynamic wetting angle. Therefore, these two mechanisms can lead to steady state de-wetting as observed experimentally- first by a high contact angles due to pollution and second by a high pressure at the cold side. The gap width and the gas pressure practically never been measured in space experiment, though, it is proposed the de-wetting phenomenon on earth with help of an excess gas pressure at the cold side [7]. Remarkable progress and impressive achievements made in detached bulk crystal growth during the last decade are compiled in the recent book [8, 9].

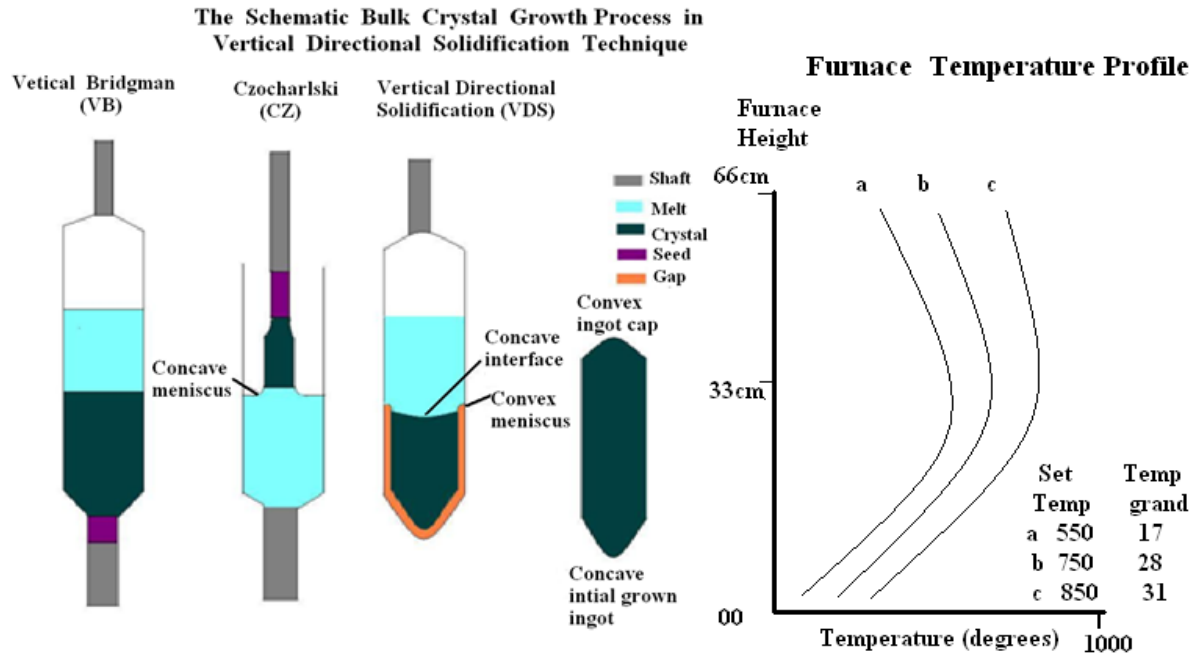

Fig.1 Schematic drawing shows the ingot growth process into Vertical Bridgman, Czocharlski and Vertical directional solidification. It also shows typical detached growth inside ampoule and its free movement for the ingot grown by VDS technique. Inset is the furnace temperature profile for different set temperature for 550, 750, 850 degrees and its respective temperature gradient.

1.3 Detached growth models

The progresses made in models of the detached crystal in last decade have been given in [5, 7, 10-12]. For detached growth, three models are considered in our research work, i.e. i) Moving Meniscus Model by Wilcox and co-workers [4, 5], ii) Capillary Model by Duffar and co-workers [5, 6], and iii) Thermo-Capillary model by Derby and co-workers [12]. The capillary model based on hydrostatic approximation of the free surface and the static meniscus is described by the Young (1805) and Laplace (1806) capillary equation. A comprehensive detached crystal growth models are cited in literature [13-24].

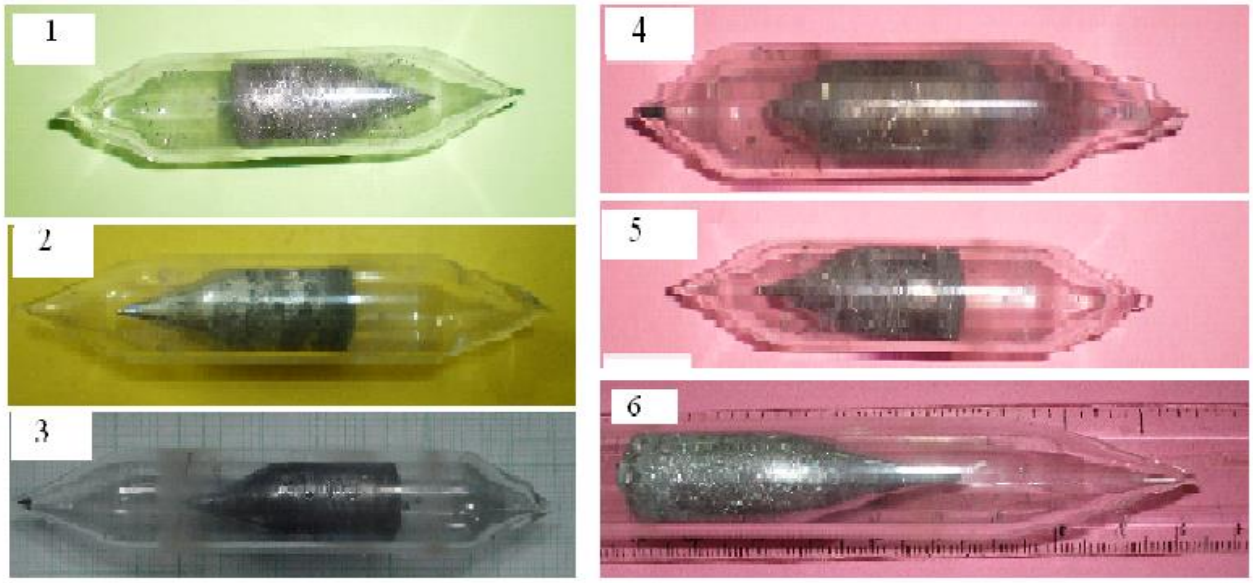

Fig.2 Typical detached ingots grown by the VDS technique: 1) InSb:Tl, 2) InSb:Bi, 3) InSb:Te, 4) GaSb:Se, 5) GaSb:Mn, 6) GaSb:In. All the ingots growth was carried out at the optimized growth conditions and parameters (see Table-1). The free movements of the ingots are seen into the sealed ampoules. The ingot is taken out from the ampoule shows the smooth and dull surfaces and the last grown shape of ingot is convex (cap) in all the detached growths. The ampoules show thin blackish oxide

layer, which acts as the encapsulation on ingot surfaces. Thus it assists to satisfy the detached condition by increase in thermal contact angle so that the detached growth occurs spontaneously into VDS growth process.

1.4 Crystal growth process in VDS

Detached growth in vertical directional solidification (VDS) technique is experiment effort for the bulk crystal growth process in terrestrial laboratory. Experimental results from 1993 have been reported in initial publishing [25] and Indian patent [26]. It showed enhancement in crystals quality: InSb [27], InSb:Te [28], InSb:Tl [29], InSb:Ga [30-31], InSb:Bi [32], InSb:N [33], GaSb [34], GaSb:Mn [35], GaSb:In [36], GaSb:Se [37] and improvement in physical properties [38].

II. EXPERIMENTAL PROCEDURE

2.1 Bulk crystal growth process

The classical methods of melt growth are Czochralski (CZ) and Vertical Bridgman (VB), see Fig.1. In crystal growth by Czochralski method, the periodic variations in impurity concentration are grown because of the rotation of the crystal, i.e. rotational striations. The closely spaced striations in both Czochralski and Bridgman-grown crystals have grown by buoyancy-driven convection. This leads to the thermal stress, which increases defects and dislocation density. To reduce the defect density in bulk materials, a modified crystal growth process is necessary. VDS technique for the growth of detached bulk crystals of high quality has been proposed. Detached growth in VDS depends on the ampoule cone geometry, filled inert gas, thermal field, capillarity effect, thermocapillary effect, self-detachment and self-pressure differences. VDS technique could be an innovative effort to grow bulk crystals of the high quality. The experimental observations into VDS have been explained similar to that used to predict the influence of gravity on detachment.

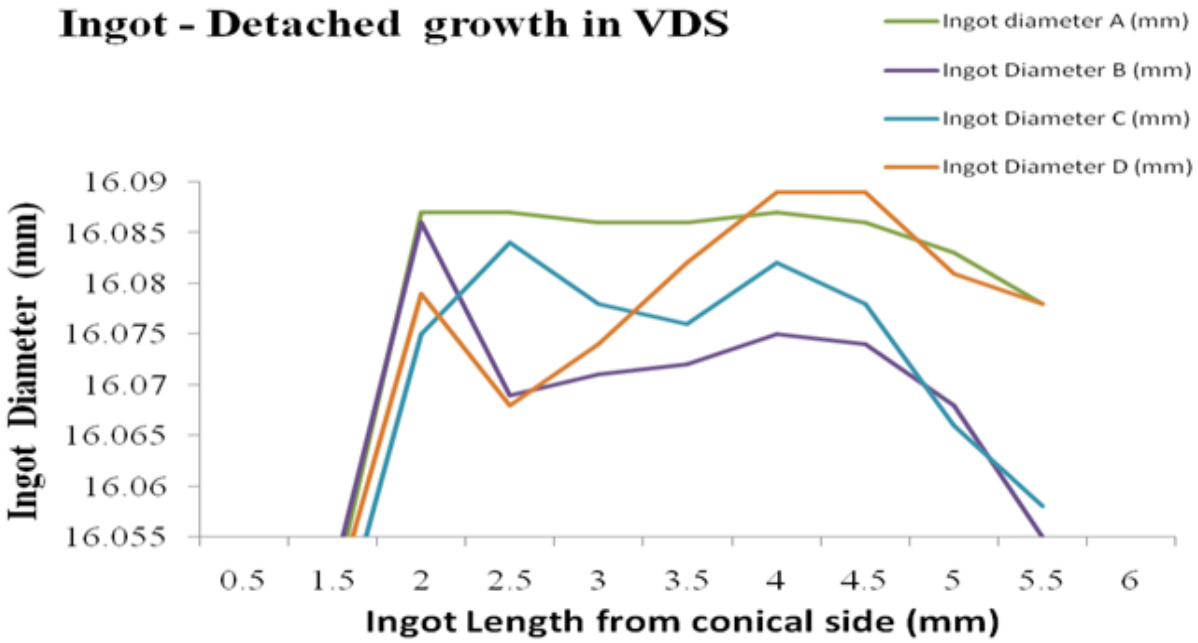

Fig.3 The graph is plotted for the ingot length against diameter of the ingot at macro level. It shows the constant gap of nearly 159 µm measured at the four different position of the ingot along the ingot axis. The gap is measurement from the ampoule ID and the ingot diameter variation position A-D (in four colours A, B, C, and D)

2.2 The crystal growth process in VDS

The source materials of high purity (5N, AlfaAeser) were used in stoichiometric proportion for the undoped and doped InSb/GaSb growth by the VDS technique [25-38]. The typical seven steps furnace temperature profile of the growth was - i) Furnace temperature was raised in 3 hours to set temperature (150^0C above m.p.), and the source materials sealed into ampoule (argon pressure 200-300 torr) were kept for congruent mixing at this temperature for 12 hours. ii) Ampoule was lowered in 3 hours to 50^0C above the melting point of the material GaSb (712^0C) and InSb (525^0C). iii) For the thermal stability temperature was maintained for 3 hours. iv) Growth parameters: growth time ≈20 hours (crystal diameter 10-22mm and length 60-65mm), the ampoule transition rates (3-7mm/h), and rotation speed (10-20 rpm). For the crystal growth process, ampoule filled with melt was slowly translated downward with the tapered end towards cold zone which acts as the spontaneous centre of nucleus formation, Fig-1. Melt freezes and self-detaches from the wall of ampoule and spontaneously self-pressure difference is developed into the gap. The solidification at low rate assists to control the heat flow and heat transfer. v) Ampoule was lowered in 3 hours to 400^0C, and vi) kept at this temperature for 3 hours. vii) Furnace set temperature was lowered to 300^0C and then switched off for natural cooling. Rotation was continued during growth at the constant speed for congruent mixing of source materials. For analysis of the detached growth, 35 InSb (Fig.2, 1-

3) and 37 GaSb (Fig.2, 4-6) ingots were grown. Experimental optimized growth parameters confirmed from the growth of 72 ingots showed in Table-1. It has been investigated that 80% ingots slide out easily from the ampoules, 15% ingots were entrapped into conical region, and 5% ingots were attached to the wall of ampoule.

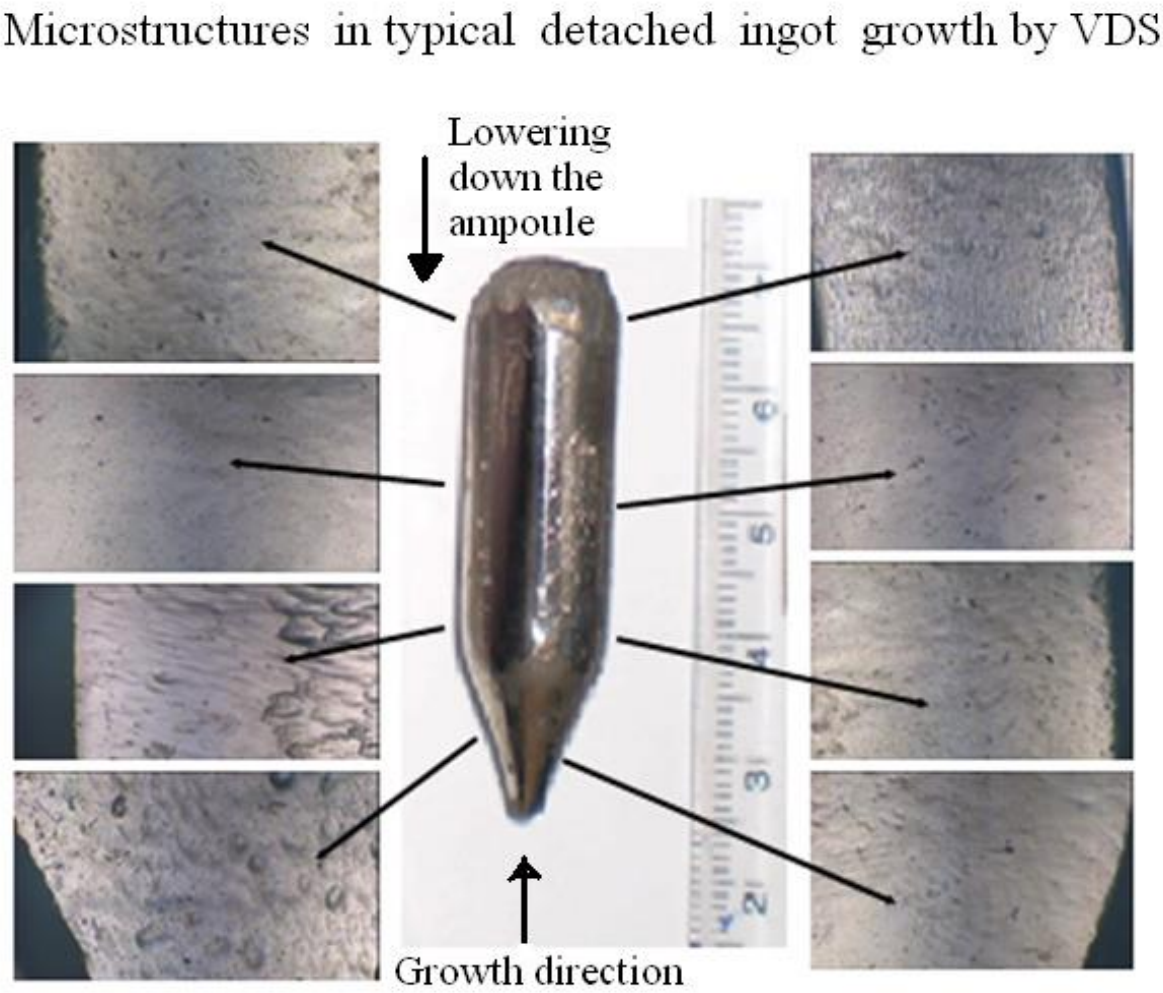

Fig-4 Growth morphology along the ingot axis for Ga doped InSb bulk crystals. It also shows the solid drop at the end due to the concentration difference. The dislocations are not seen at the edge of the periphery of the samples. There are tiny voids are seen due to the presence of bubbles in some growth.

2.3 Detached growth process in VDS

In the growth process, furnace setting temperature is 150^0C above the melting point of the source material with the maximum melting point and maintained constant throughout the ingot growth, so that crystal-melt boundary is steady and stable at the melting point. Steady and stable axial temperature gradients at difference set temperatures showed in Fig-1, and radial temperature gradient is 2.5^0C/cm. There is no temperature fluctuation as it is controlled by PID controller with variation +1 or -1 degree. The presence of argon gas stimulates to process of the spontaneous pressure difference into the gap which helps for the detached growth. The argon gas, which was trapped at the bottom of conical ampoule shape at the beginning growth process, did not balance the sum of hydrostatic melt pressure, and the weight of the melt on the meniscus. But after reduction in the melt height the argon pressure did balance the mentioned sum by meniscus surface tension effect, and the detached growths originate into the tapered ampoule. This argon gas pushes melt away from the wall of ampoule, thus the free surface/meniscus forms. In the process of lowering the melt filled ampoule from high temperature to low temperature region of the furnace, when its tip reaches just below its melting point, the free molecules of tiny melt freezes away from the ampoule wall at the beginning and forms solid. This is the first step of detached ingots growth which acts as the seed, i.e. growth without a seed. At this position the preferential orientation growth of ingot is promoted spontaneously. This process is continuously propagated till growth complete. Thus a gap between the freezing solid and wall of the ampoule arises spontaneously by the existence of wide free surface area Fig-2. It is the balance between gravity, surface tension and liquid pressure which can result in the displacement of a fluid in narrow capillary tubes in which displacement depend on surface tension and geometry of the ampoule.

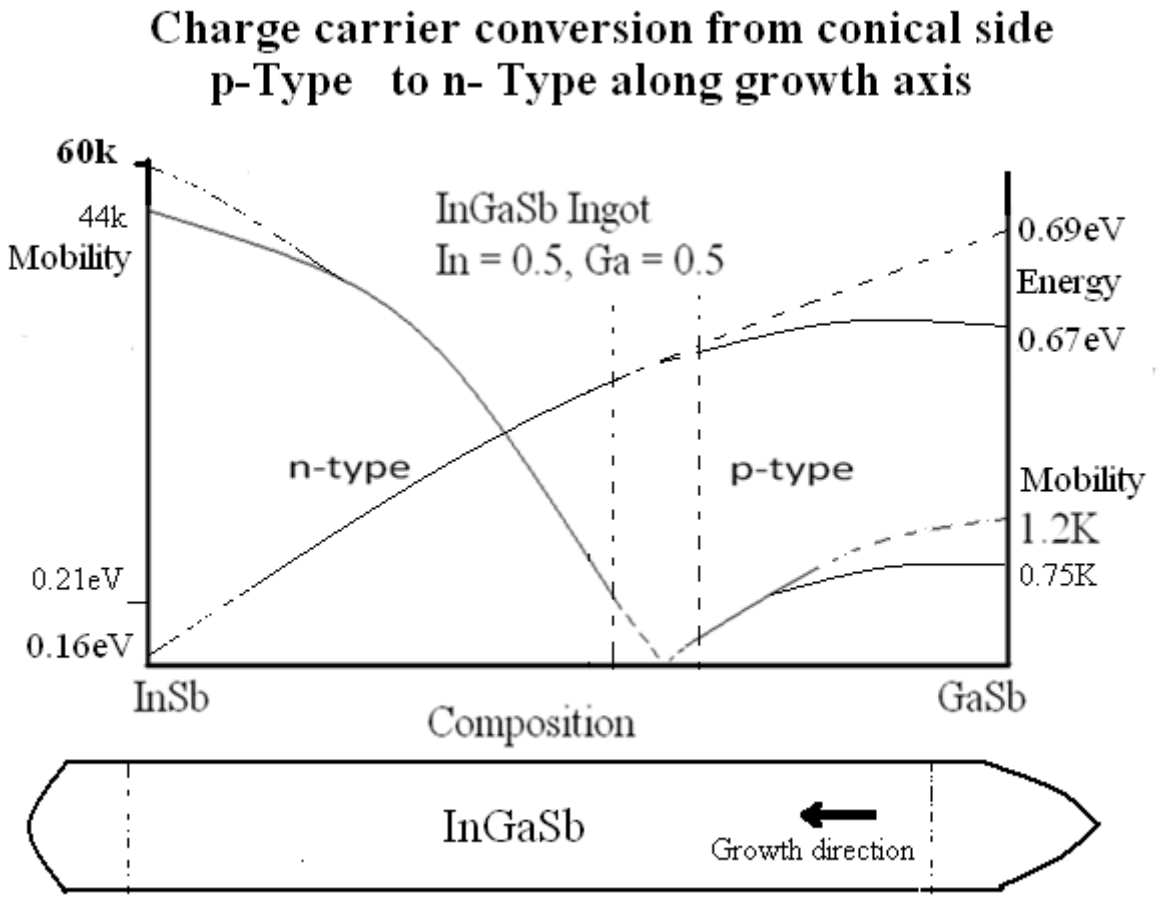

Fig-5 The physical property variation along the ingots growth axis for the Ga doped InSb ingots. The ingots showed the conversion of charge carrier from p-type to n-type along the growth direction from tapered end of the ingots. The melting point of GaSb is higher than the InSb, therefore, near conical side freeze growth showed the GaSb solidification. Indium (In) incorporation increase as the growth proceeds and finally the concentration of InSb freezes. Dotted lines are for pure binary material of InSb and GaSb.

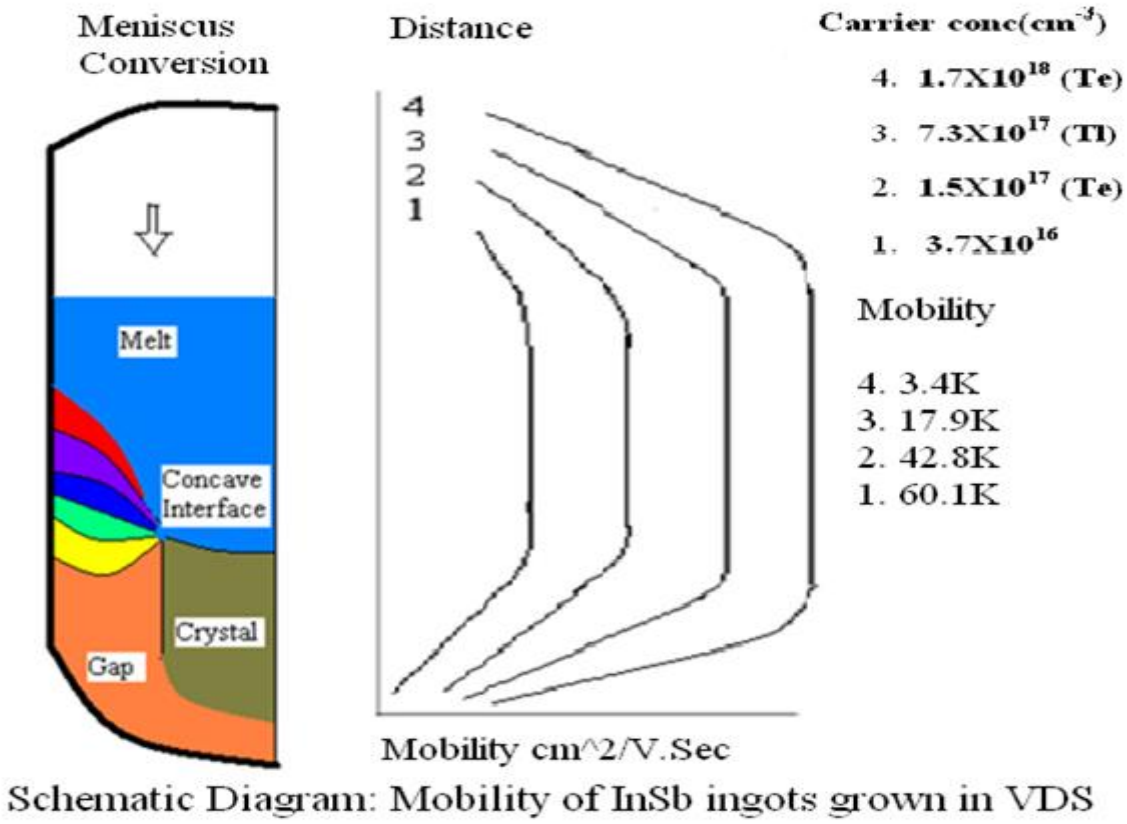

Fig-6 Experimental results of the carrier concentration and mobility, and their correlation with the conversion of meniscus from concave to convex (yellow to red colors) at the steady and stable interface is shown in above diagram. At the ingots end there is convex cap (seen from top). The interface concavity also decrease along the growth axis. The nature of Hall measurements along the growth axis is shown in graph for mobility versus growth distance. All detached ingots grown by VDS showed similar nature. The number one represent for the undoped InSb ingots growth while other numbers are for doped growths. Doping decreases the mobility of ingots. Similar trend of growth is also observed for GaSb ingots growth.

2.3 Characterization of the as grown ingots

The ingots were cut perpendicular to the growth direction to obtain 500μm thick substrates and then the polished (sample dimension 5x10x10mm^3) were used for the analysis. The substrates surfaces were cleaned with usual cleaning methods. For the growth morphology, samples of (220) orientation were etched by CP4 and modified CP4 solutions for the etch-pit density (EPD). Surface defects were identified with Scanning Electron Microscopy (SEM) and composition by Energy dispersion analysis by X-Ray (EDAX). The crystals quality and lattice parameters were determined by the X-ray diffractometer (XRD). Energy gap is determined by Fourier Transform Infrared (FTIR) spectroscopy. Raman spectroscopy and Micro-hardness were studied for quality identifications. Hall - van der Pauw measurements used for the electro-physical properties. In all ingots, the last frozen melt showed the convex shape, i.e. cap of ingots. Some caps with the off-white band and some caps with blackish band near the ingot cap.

III. RESULTS AND DISCUSSION

3.1 Diameter measurement of the detached ingots

Detached growths under terrestrial conditions in VDS technique have been accomplished (experienced since 1993). In these experiments, it is investigated that the ingots come out of the ampoule easily as diameters of the ingots were smaller than the inner diameter of the ampoule by differential thermal dilatation Fig-2, 3. It also shows gap width variation, and its surfaces were dull and melt did not sticking to the wall of ampoule Fig-2, 4. These ingots were grown without the seed, without contact to the ampoule wall, without ampoule coating, and without the external pressure. It is investigated [25-38] that the as grown ingot moves freely inside the ampoule and it is repeatable and reproducible. Thus the term detached growth is used. In VDS, three gap growths are investigated i) a gap constant, ii) a gap decreases and iii) a gap increases

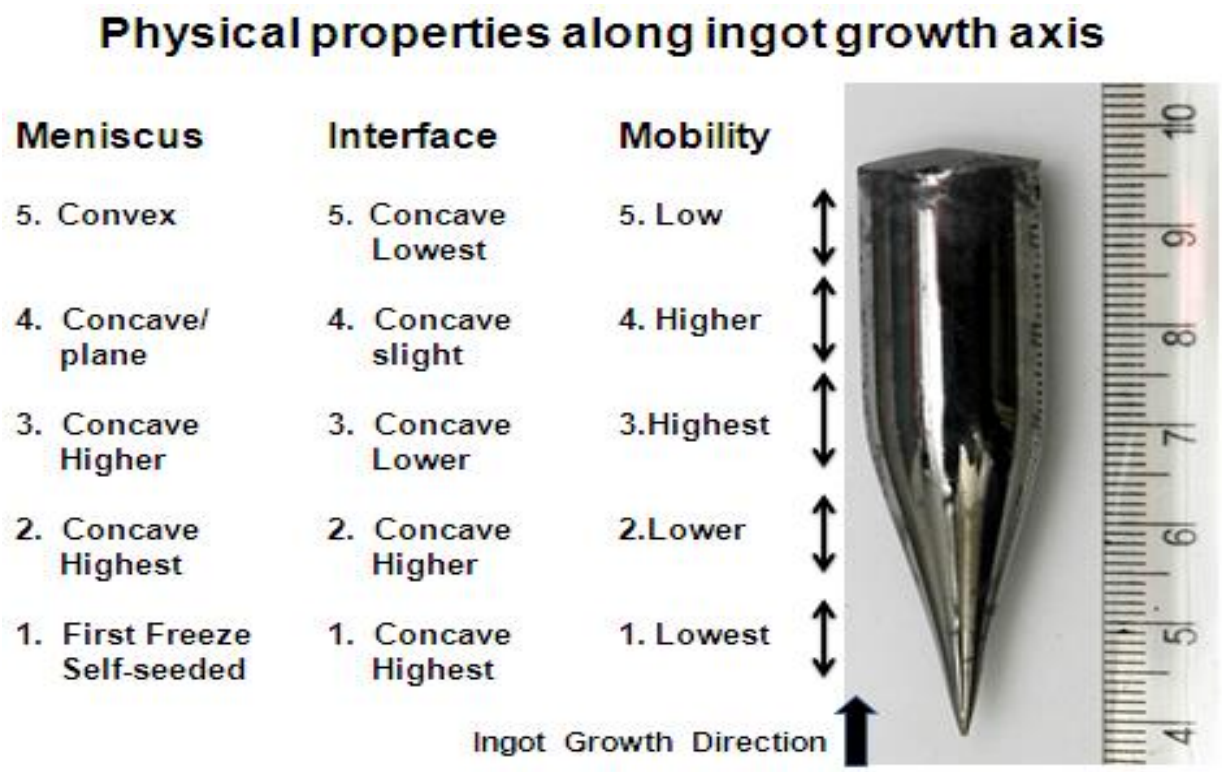

Fig-7 The measured physical properties along the ingots growth and its relation with the meniscus and interface shape. It is corelated with the experimental measured mobility along the growth axis and then predicted the shape of meniscus and interface on the bases of crystal quality and experimental result. Its comparision with physical properties is considered in Fig-6. Pyhsical properties enhancement are also published in our references.

3.2 Growth morphology

The analysis of the doped InSb/Gasb ingots showed the variation in doping < 10% and dislocation density is 10^5 cm^2 at the initial growth (tapered end) then it is decreased along the crystal axis, it reaches $<10^3$ cm^2 in the middle of the ingot and then slightly increases towards ingot cap. In detachment, the dislocation and defects are not grown at the edge of the substrate but the growth pattern is grown at the centre in some ingots. The growth morphology of $In_{0.5}Ga_{0.5}Sb$ along the crystal axis is shown in Fig-4. It shows improved crystallanity of ingots due to the large grain size and increased in this size after the shoulder of the ingots.

3.3 Physical measurements

The enhancement in physical properties of crystals grown in VDS technique (reported in our references) has been confirmed by SEM, EDAX, XRD, FTIR, Raman spectroscopy, and Vicker microhardness test measurements, and these results have been published elsewhere [38]. Hall van der Pauw measurements showed the interesting conversion of charge carriers from p-type to n-type along growth direction for the $In_{0.5}Ga_{0.5}Sb$ bulk ingot growth as shown in Fig-5.

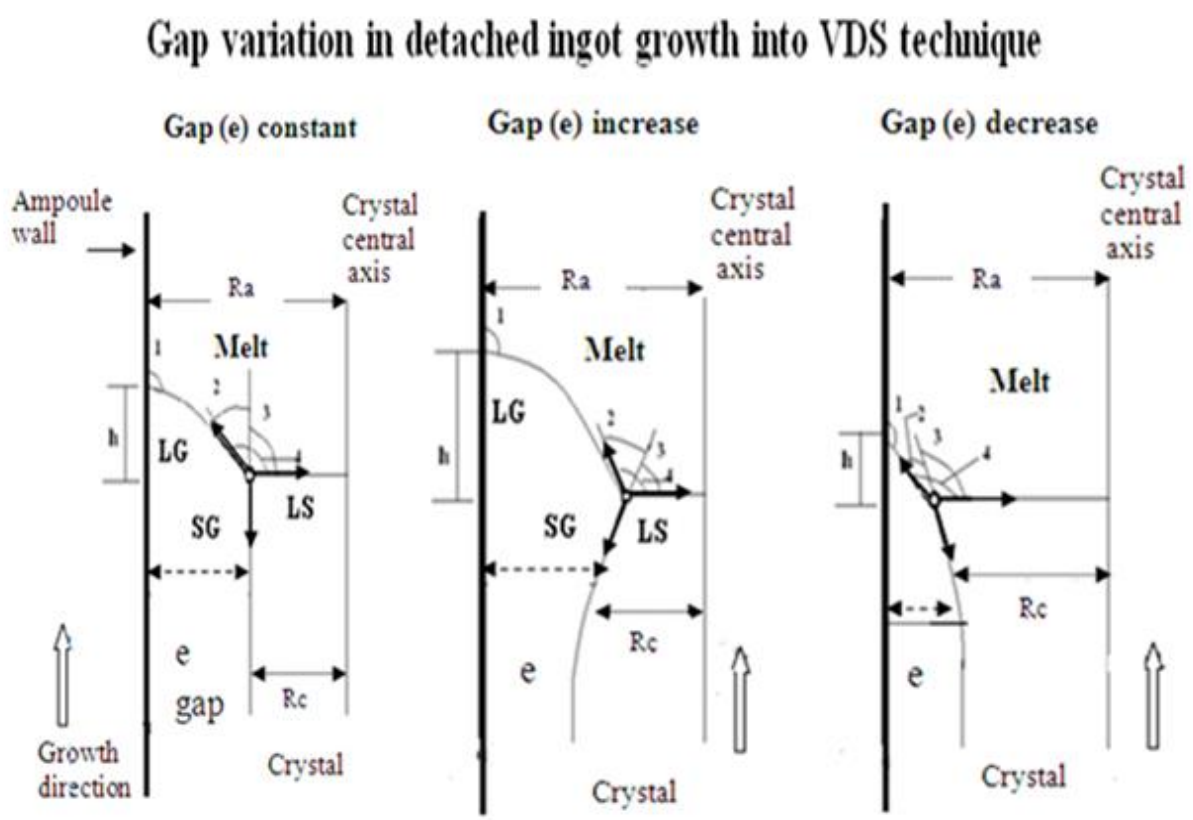

Fig-8 Enlarged sections of tirade (triple point O, hollow white dot in joint of dark arrows). The three process of gap variation are predicted on the basis of experimental measured gap width. Pm: pressure above melt, Pgap: pressure in gap, 1: Θ_c- equilibrium contact angle, Θ_d: dynamic contact angle, 2: α – Ingot growth angle, 3:Θ: Angle (in melt) between SG and LS, 4: ψ - Angle into melt between LG and LS. $\Psi = \Theta + \alpha$, this relation depends on the movement of phase frame. The upward arrow indicates the growth direction. The bold straight dark line at left of each diagram represents the outer surface of the ampoule. For constant triple vector, gap is constant, if these vectors moves inside the melt then gap increases while it moves towards the wall of the ampoule it result in decrease in gap.

IV. THE PHYSICS BEHIND DETACHED GROWTH

Since for four decades detached growth is observed in microgravity gravity experiment four decade earlier and it could explore the new outline of the crystal growth science and technology. However, the initiation of detached growth mechanism is yet comprehends inadequately. The molecular interactions and interfacial phenomenon are not influenced by the gravity. Reduction of melt flows in microgravity and reduced gravity could help to differentiate the bulk surface phenomenon, and the quantitative knowledge in terrestrial growth technology is in progress. Unfortunately, none of the models cited concur with the correlation to published VDS experimental results as VDS some growth parameters differs with that modeling data.

4.1 Stable and steady meniscus and concave interface position

At the position of melting point of materials, there is a melt above the interface and solidified crystal below it. Growth process is at the stable and steady – meniscus with concave crystal-melt interface, Fig.6. Evidence of undoped and doped InSb/GaSb detached ingots based on Hall measurements, carrier concentration and mobility, a model is predicted for meniscus conversion from concave to convex shape along the crystal axis (yellow to red color) at steady position Fig-6. Hall mobility along crystal axis and its statistical analysis is used to predict a model for mobility relation with the meniscus and interface along the crystal growth axis, Fig-7. The physics of detached crystal growth process in VDS experiment is explained and the process of gap constant, gap increase and gap decreased showed in Fig-8.

Table-1 Experimental optimized growth parameters

In this table, optimized data has been confirmed by the seventy two experiments of InSb and GaSb ingot growths by VDS. It has been shown the experimental evidences for the emergence of the detached growth and gap formation.

Growth data	Growth rate mm/h	Rota-tion rate rpm	Tempe-gradient Axial ^{O}C/cm	Tempe-gradient Radial ^{O}C/cm	Filled gas pressure Torr	Cone angle $^{0}\theta$	Growth period hrs	Conve-xity ingots mm
Optimum growth value	3-10	10-15	15-35	2.5	200-300	55-75	65-70	1- 2

Table.1: The optimizations of crystal growth parameters have been arrived after the growth of undoped and doped for the InSb, and GaSb growths in above table. From these parameter ranges, we have performed seventy two growths out of these ingots several ingots showed detached growth for entire ingot with enhancement in crystallography and physical properties [25-38]. For detached growth in VDS, there are growth condition such as interface position into furnace should be steady and stable state, interface always concave with respect to the melt, melt height decreases with reference to up-word crystal growth, no fluctuation into the set temperature, temperature gradients of the set temperature should stable, no fluctuation into the growth rate and the filled argon should be of <20ppm of oxygen contains.

The meniscus shape is unstable when $\theta_c + \alpha < \pi$, and stable when $\theta_c + \alpha > \pi$, θc: Young thermal contact angle, α_0: equilibrium growth angle, α: growth angle during ingot process [20, 21]. This is in good agreement with VDS grown InSb and GaSb ingots. The typical gap constant of nearly 159μm showed in Fig-3. The higher surface tension gradient moves towards the lower surface tension gradient from the meniscus to the interface in the melt with the growth morphology away from edge of the ingots, Fig-4. Similar to a thermocapillary model for temperature-dependent surface tension effects at the melt menisci, the gap variation shows signs of strong free surface that extends into the melt. All detached growth showed the decrease in a gap near convex cap (increase in crystal radius) and the cap of ingot covered with blackish color. The gap width predicted by models cited in literature and that shown by VDS experiments are in good agreement. The variation in diameter versus ingot length at micro level at four different places beyond cone is plotted along the ingot. The variation in diameter is of the order of~18μm along ingot axis (similar to profilometer). This is due to the changes in dynamic pressure Fig.3.

V. CONCLUSION

Overall statistical analysis of experimental result of the crystal growth process by VDS is reported in our references. The measured three gaps and prediction based on the gap variation are relevant to the experimental data. In terrestrial laboratory conditions, the pressure difference is changed during the growth to counter balance the difference in static pressure originate by the decrease in melt height as progress in ingot growth. Forces at meniscus are in balance when the sum of static capillary forces and the melt weight is equal to the self pressure difference, which is a necessary condition to be satisfied into VDS technique to exist the gap. Self controlling pressure difference and self detachment as a function of gap width in the detached growth has been emerged. Crystal growth by VDS is reproducible, reliable and ensures the enhancement in crystal quality with high yield and seems to be more profitable for commercial method.

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AUTHORS

First Author – D. B. Gadkari, Department of Physics Mithibai College Mumbai-400056 India, E-mail: dr_gadkari@yahoo.com; db.gadkari@gmail.com