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Detached/Contactless Growths, Reduced Melt Convection and Its Effect on the Device Grade Substrates of SB-Based Crystals Grown By

# **VDS on Earth**

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**ABSTRACT:** Vertical directional solidification (VDS) under the controlled growth conditions and parameters has been operated to solidify antimonide (Sb) based bulk crystals for the entire detached/contactless growths. In ground experiments, Crystal qualities have been studied in VDS, under the effect of reduced melt convection. Recent detached crystals grown by VDS have been studied for quality identifications:  $InSb_{1-x}Bi_x$ ,  $InSb_{1-x}N_x$ ,  $GaSb_{1-x}Se_x$ , and  $GaSb_{1-x}Mn_x$ . Detachment is a key factor for the dopant distribution in microhomogeneities under terrestrial condition. Dopant distributions along the transverse and longitudinal axis of entire detached ingots have been measured. Crystal properties had confirmed that the homogeneous substrates, higher structural quality and reduction in convection into melt, at the macro and micro level. VDS crystal properties are analogous to the crystals grown in microgravity. Methodology of solidification seems to be the promising technology for the device grade bulk crystals. VDS crystal growth system has prospects as the commercially profitable system.

**KEYWORDS:** A1: Directional solidification, A1: Convection, A1: Doping, A2: Growth from melt, A2: Detached growth, A2: Microgravity, B1: Antimonides, B1: Crystal growth, B2: Semiconductors III-V

### I. INTRODUCTION

Progress of space technology has led to the development of the materials science, a new field of science and technology under microgravity conditions. Space experiments have been reviewed in "Crystallization in Space: Results and Prospects- Crystallography Reports-2014" by Strelov-Voloshin-et.al. [1]. Microgravity is a key factor in concluding microinhomogeneity of a dopant in the grown crystals, the high electrophysical properties, and negative effect of an ampoule. Volosin et.al, [2] has described the cause of unexpected radical progress in detached part of space grown crystal related to the free melt surface and contactless growth. Space programs NASA-Skylab spacecraft-1974 (USA) [3-5] and Apollo-Soyuz spacecraft-1975 (USSR) [6] have performed the extensive experiments to enlighten a new concept of detachment; however, the experiments are expensive. Though more than 800 experiments in spacecraft have been carried out, researchers have not obtained space crystals with the entire detached growth. Hence, required condition for the detachment on earth is a big challenge. On the other hand, it conveys a new fundamental knowledge and strengthens the science and advanced technologies. Wilcox, and Regel [7] model was based on the force balance between surface tension and gas pressure in the gap separating the melt and ampoule. Under low gravity conditions gas present in system contribute to separation of the crystal and melt to facilitate contactless growth, and out gassing mechanism are suitable for closed systems. Wang-Wilcox et.al [8] in their first experiment of detached growth of InSb on earth in 2004 observed that the phenomenon is complex due to the hydrostatic pressure, the existence of convections, and tried to minimize these both by a furnace in which temperature



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increases with the height. Duffer et al. [9] proposed that, surface roughness and growth angle of the ampoule were the primary reasons for dewetting; and developed amethod to set up pressure difference between upper and lower menisci. Duffer and co-workers' [10] employed a method by which gas was injected into upper and lower vapor spaces of the ampoule.

Yeckel-Derby and co-workers' in thermocapillary model coupled the proportional-integral feedback control to stabilize a detached crystal growth using shape stability. The melt in the gap is supported by a liquid meniscus that connects the triple-phase line (TPL) to the wall [11, 12], and references are listed since 1975. Cröll -Volz et.al [13] reported that, detached growth was found 40 years back in microgravity experiments. However, the reasons for the detachment are not investigated yet. Binetti- Cröll et al [14] studied the effect of a reduced melt convection in growth process and its effect on the ingot quality of silicon (Si). In space experiment of GaSb:Bi, Danilewsky-Croell et.al [15] observed that the thermocapillary convection and the surface tension influence presence of the curved dislocations. The dislocation densities within 1–2mm at the edges of crystals were witnessed. Volz-Mazuruk [16] has studied the existence and shapes of menisci in detached Bridgman growth. Recently, Sylla-Duffer [17] has performed study of solid-liquid interface and meniscus in growth of GaSb and InSb. In their theoretical models, stable dewetting was obtained for GaSb for an oxidizing atmosphere present in the ampoule, however, the stable dewetting of InSb was never obtained [18]. Reports in [17] are i) the hydrostatic pressure elimination will be not possible on earth, and then detachment in a soft ampoule would be impossible. ii) Several atmospheres were applied to study the effect of oxygen on the detachment. However with neutral or reducing atmospheres, it is possible to force meniscus beneath stable melt conditions but it did not slid along the crucible wall and the growth was always attached. iii) In GaSb experiments, detachment was observed only during oxidizing atmosphere. iv) Efforts have been made in order to form the stabile meniscus in InSb growth but stable detachment of InSb has never been obtained, whatever the atmosphere used. Sylla-Duffer et.al [19] observed that the space grown detached crystal region exhibit dislocation levels up to two orders of magnitude less than earth grown crystals and free of twins. Epure-Duffer et. al [20] dewetting cause on earth is the self-stabilizing gas pressure difference. Braescu [21] showed pressure difference with specific gap size of crystals grown in terrestrial dewetteing. Lyubimova et.al [22] mathematical model accounts a crystal detachment from the ampoule wall.

Prostomolotov-Voloshin et.al [23] have investigated thermogravitational convection and impurity transfer in the melt using a numerical model for GaSb: Te by Bridgman in the microgravity conditions. Serebryakov et.al [24] had performed growth process on earth in the vertical Bridgman method under conditions of the weakened thermogravitational convection. On Earth, vertical method with axisymmetric heating enables a 2-3 orders of magnitude decrease in the level of thermogravitational convection [25]. Serebryakov showed that the spreading resistance distribution reveals the heat and mass transfer processes in the melt. In GaSb:Te, the concentrations of the donors  $(N_d)$  and acceptors  $(N_a)$  were nearly equal. The increased sensitivity charge carriers are due to a shift in the donor-acceptor ratio in the process of heat- and mass-transfer. Strelov-Voloshin et.al [26] proposed methodology of carrying out terrestrial experiments, in which the thermal gravitational convection intensity in melt was reduced by two to three orders of magnitude. Sakata-Hayakawa co-workers [27] have grown crystal in a space and ground condition, and to understand growth mechanism of In<sub>x</sub>Ga<sub>1-x</sub>Sb crystal. Nobeoka-Dost et al [28], simulation result of InGaSb grown crystals showed that under space and terrestrial condition, diffusion was dominant in the growth process. PENG et.al [29] described that the magnetic field can effectively suppress melt flow and thermocapillary fluctuations. LI- PENG et.al [30] model, analysis of the buoyant-thermocapillary (Marangoni) convection during detached solidification has effect on a gap width and melts height. Recently, Inatomi-Hayakawa et al [31] gave an account of the experimental results of  $In_xG_{1-x}Sb$  alloy at the International Space Station (ISS) and terrestrial experiments. Important point to be noted is sample quality of  $\mu g$  (g-gravity) is better than the gravity (1g). Cröll-Volz [13], theoretical investigations and new experiments have shown detached growth can be achieved on an earth. Microgravity environment is a tool for investigation of the crystal growth processes to improve quality of crystals on Earth. Yet, the mechanism of detached growth is not fully understood. Space grown detached crystals are required in microelectronics [32], optoelectronics [33], microwave techniques [34], emitters [35], design of new generation laser [36] and radiation detector [37].



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The solidification process of crystal growth in a vertical directional solidification (VDS) is designed to combine advantages of both the Czocharlski and vertical Bridgman method, i.e. combination of the growth principals'. In VDS since 1993, a specially designed vertical furnace had been used for the analysis of 72 grown ingots. The VDS might insensitive to thermogravitational, thermocapillary, and the Marangoni convection, and the effect of an ampoule wall. Thus detached growth in VDS may be analogous to that of the space grown crystals, but it needs analysis. Experimental results of the detached process in the terrestrial laboratory are published elsewhere [38], Indian patent [39], enhancement in quality: InSb [40], InSb:Te [41], InSb:Tl [42], InSb:Ga [43-44], InSb:Bi [45], InSb:N [46], GaSb [47], GaSb:Mn [48], GaSb:In [49], GaSb:Se [50], improved physical properties [51-52], and devices with low dark current [53-54]. The higher sensitive substrates have potential to fulfill technology for advanced applications. Reproducible, contactless, high quality ingots and substrate properties suitable for devices have been reported.

#### **II. EXPERIMENTAL PROCEDURE**

#### **II.1 AMPOULE PREPARATION AND SOURCE MATERIALS FILLING**

A quartz ampoule was designed to form conical shape / tapered end to manipulate the detached growth in VDS on an earth.. Various dimensions of the ampoule are: conical section ~ 20-25mm long, cone angle of the order  $55^{0}$ , ingot length ~65-75mm, diameter 10-24mm and ampoule length ~100mm. The ampoules were cleaned, rinsed repeatedly with acetone; methanol, deionized water followed by high temperature flames cleaning for out gassing. Source materials (In. Ga, Sb) and dopants (Bi, N, Se, Mn) of high-purity grade (5-6N) had been purchased from AlfaAesar (Johan Matthey Com). Materials were ground a particles size diameter < 2mm and were loaded into ampoule. Ampoule was alternately filled and re-evacuated by the argon gas (pure, industrial, and commercial) ten times in vacuum (~10<sup>-5</sup>torr). Finally the source materials and an argon gas (200-300 torr) was sealed. The source materials: InSb, and GaSb (stoicometric proportion) with dopants were filled to achieve ingot length ~ 65-75% of the ampoule length. Syntheses of ingots were performed under an argon atmosphere to prevent oxidation.



Vertical directional solidification (VDS) system

b) Furnace photo graph used for growth

Fig-1 a) The schematic diagram of the VDS, b) Photograph of VDS, c) as grown GaSb:Mn ingot into ampoule, and d) The free movement of ingot inside ampoule. Numbers in figures-1) quartz growth chamber (L=100cm, dia=10cm), 2) Wilson seal, 3) Vertical furnace with the thermocouples, 4) vertical shaft to hold the ampoule into centre of furnace, 5) quartz ampoule, 6) clockwise and anti-clock wise rotation for ampoule, 7) up and down movement of ampoule, 8) as grown ingot inside ampoule, 9) Open space above melt field with argon pressure, 10) Quartz ampoule.

a) Schematic VDS diagram



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#### **II.2 FURNACE DESIGN AND TEMPERATURE PROFILE**

Fig-1 shows: a) schematic diagram, b) photograph of VDS and c, d) GaSb:Mn detached ingot. Fig-2 shows- I) Furnace temperature profile, II) Furnace photograph, and III-A, B, C) the schematic detached/contactless growth inside an ampoule, IV) detached ingot and V) attached ingots. III-A) initial growth, the nucleation growth of the melt with concave meniscus and concave interface have seen from the melt top. III-B) middle growth, the straight meniscus and concave interface, and III-C) end growth with the convex meniscus and the reduced concave interface. A single zone furnace was used to obtain a temperature profile around the ampoule to grow crystals. The quartz tube at the centre of furnace (length -100cm, diameter -10 cm) and hot zone (growth chamber  $\sim 1000^{\circ}$ C) showed schematically in Fig-1a. It consists of single resistive heating zone, and the heater with thermocouples controlled by the PID for stable thermal conditions to prevent the temperature fluctuations ( $(\pm 1^{0}C)$ ). The heater has axial temperature zone (flat)  $\sim 10$  cm in length along the quartz tube with a maximum setting temperature  $1000^{\circ}$ C at the centre. The high axial temperature gradient in the furnace (25-35°C/cm) and low radial temperature gradients (1.5- $2.5^{\circ}$  C/cm) for the respective setting temperatures ( $550^{\circ}$ C-900<sup>o</sup>C) were measured by the thermocouples. In this investigation, instead of moving the furnace, we moved ampoule downward from the set temperature of the furnace by keeping steady and stable three phases (liquid, solid and gaseous) at the juncture of the interfaces (liquid-solid (LS), liquid-gas (LG) and solid-gas (SG) in the fixed position in contrast [8,10]. The pulling down, and rotation rates were 3-7mm/h and 10-15 rpm respectively. Lower solidification rate assists to control the heat flow and heat transfer. Ampoule position and thermal profile is shown in Fig-2. In contrast to the conventional growth, spontaneously grown self-seed was acted as the seed in VDS experiments. Experiments were always carried out in a back filled pure argon pressure, which found mandatory to maintain stable detached growth.



Fig-2 I) Temperature profile of the furnace for set temperatures (550°C, 700°C, 850°C), II) Photographed of isolated furnace, III) The detached ingot's conversation mechanism of meniscus and interface, i.e. A, B and C), IV) The detached InSb:Bi ingot out of the ampoule, V) Attached InSb:N ingot inside the ampoule, which showed the degraded crystal qualities.

#### **II.3 THE SEVEN STEPS DETACHED GROWTH PROCESS**

In brief, ampoule with the source materials was positioned vertically in a hot zone with the conical tip of the ampoule in the flat zone Fig-2II, orange line, (detail Ref-38-54). Typical seven steps furnace temperature profile was



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employed: i) Furnace temperature was raised in 3 hours ~150°C above the melting point of source materials and maintained for mixing and melting the materials congruently (homogeneous) for 12 hours. ii) Ampoule was lowered in 3 hours to a temperature 50°C above the melting point (InSb-525°C, GaSb-712°C). iii) This condition was maintained for 3 hours for thermal stability. iv) Growth parameters: temperature gradients (axial, radial), the downward transition rate (5mm/h), and rotation speed (10rpm) were continually applied. No downward support to an ampoule to prevent heat conduction. In growth downward process, ampoule filled with melt was slowly translated downward with the tapered end towards cold zone, which acts as a spontaneous centre of nucleation. v) Ampoule was lowered in 3 hours to  $100^{\circ}$ C below the m.p. and vi) kept at this temperature for another 3 hours for thermal stabilization. vii) Furnace set temperature was lowered to  $300^{\circ}$ C and then switched off for natural cooling. While removing the ingots, the detached ingots came out easily by gently tapping the ampoule (InSb:Bi) Fig-2IV, but in attached growth, the ampoule had to be broken (InSb:N) Fig-2V). In attached growth, intensely argon was not refilled into an ampoule. Experimentally optimized growth parameters and conditions have been confirmed from the growths of 72 ingots (InSb, GaSb, alloyed and doped) since 1993. It was investigated that i) 80% ingots slide out easily from the ampoules, 15% ingots entrapped into conical region, and 5% ingots attached to the wall of ampoule. Our analyses reinforce the concept that favorable thermal conditions, parameters, and argon pressure inside an ampoule influence the detached growth in VDS on an earth. The crystallization process in the furnace is vertical mode by moving the ampoule relative to the heaters (furnace position fixed). In sealed ampoule design, the source material melt was in closed volume and there was no free melt surface. This facilitates reducing thermogravitational (Rayleigh) convection, and maintains the axial symmetry of the thermal field. On Earth, vertical method with axisymmetric heating and closed ampoule both sides, decreases the level of thermogravitational convection by 2-3 orders of magnitude. This process attributed to microgravity [24-25].

### Growth morphology in attached ingots



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Growth morphology in detached ingots

Fig-3 Growth morphology in detached and attached ingots, in attached growth substrate cut transverse to the growth direction, while in detached substrate cut longitudinally to the vertical growth axis.

### **II.4 THE SUBSTRATES PREPARATION**

The substrates were cut transverse to the growth axis and then mirror-polished using alumina abrasives powder, and were cleaned with distilled water and acetone. Substrates  $10x10 \times 0.3$ mm<sup>3</sup> were used for the analysis. Our



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conclusion is that the contactless Sb-based substrate surfaces become smooth, mirror finished easily, in contrasts to the attached growth.

#### **II.5 MEASUREMENT AND CHARACTERIZATIONS**

Growth morphology and etch-pit density (EPD), substrates [220] etched by CP4 and modified CP4 solutions (HF: HN03: CH3COOH (1:3:3 and 1:3:3:10H2O), were studied respectively. The surface morphology was studied by the Nomarski and Karl Zeiss Jean microscopy. Hall measurements by the van der Pauw technique were used for the electro-physical properties. Surface defects were identified with Scanning Electron Microscopy (SEM) and composition by Energy dispersion analysis by X-Ray (EDAX). The growth direction was determined by Laue X-ray back diffraction. The crystal quality and lattice parameters were determined by the X-ray diffractometer (XRD). Energy gap was determined by Fourier Transform Infrared (FT-IR). Raman spectroscopy patterns were obtained for the orientation and quality identifications, and microhardness studied by Vickers test. EBSD and SIMS were employed for the defects, orientation and compositional analysis.

#### **III. RESULTS AND DISCUSSION**

#### **III.1 PRELIMINARY OBSERVATION OF THE GROWN INGOTS**

Entire detached ingots grown by VDS experiments are without micro features on surfaces. In this research, recent typical ingots grown have been described. Fig-1 shows entire detached GaSb:Mn ingot with free movement into the ampoule. Growth experiments in VDS confirmed that the pure argon gas pressure inside ampoule is mandatory for the entire/complete detachment. Detached and attached ingots apparently differ in surface features. For detached growth, the dull surfaces, convex shape (cap), and convexity of the ingots cap vary from flat to high convexcity (~2.4mm) showed Fig-1c, Fig-2IV. The maximum convexity was measured for the lower ingot diameter. But unlike the partial detached crystal, surfaces in microgravity were attributed to the striations, distinctive ridges touching crucible, forming narrow grown islands or hillocks. Noticeable detached growth, VDS ingots differ oxide proposal, which forms scum on melt-gas interface and hinders surface smoothness.



Fig-4 EBSD a) IQ mapping of the InSb:Bi detached ingots shows very large single crystal region, b) color code map for orientation of the crystal, and c) inverse pole figure of InSb:Bi



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#### **III.2 DETACHED GROWTH IN VDS**

#### **III.2.1 SELF- SEED GROWTH**

Since 1993, we have been grown the detached ingots in VDS [39]. However, the design of ampoule tapered end, back filling of argon gas before sealing, the process of nucleation, gap thickness, thin layer of oxide formation in some growths, conversion of meniscus from concave to convex and three gap variations were investigated and optimized for detached crystal growth [39-42, 47-49, 51-53].

Features of the detached growths are observed in recently grown seventeen crystals: InSb:Bi (7), InSb:N (4), GaSb:Se (3), GaSb:Mn (3), (number in bracket indicate number of growth). These ingots have been grown by VDS with smooth quartz ampoule under pure argon gas pressure, but some growths showed thin oxide layer due to the presence of impurity (source materials). Detached growth by VDS has observations, which shows the inconsistency, and differ the model analysis reported as in above [17]. On the basis of detached ingots growth in VDS, experimental results and previous explanation in our references, we would like to propose a new explanation of detached growth in VDS based on the free melt surface similar to the model proposed by Volosin et,al [2] for space grown GaSb:Te. VDS results are in good agreement and analogous to the results reported by Volosin, which adhere the '*laws of physics are similar in the same inertial frame' and the explanation could be used for the terrestrial detached growth experiments* 

#### **III.2.2 DETACHED GROWTH MECHANISM AND MENISCUS CONVERSION**

In VDS technique, a furnace had been designed with axisymmetric supply of heat to melt source materials in the ampoule (Fig-2), and Seven steps growth profile (section II.3). *If thermogravitational convection* [1,2,26] *is eliminated in the melt, then conditions are close to that of diffusion mass transfer.* The concept is in agreement with the detached ingots grown by VDS technique.

During the crystal growth, when a tiny melt (dangling) in conical tip of an ampoule first enters below m.p. of the melt, nucleation is spontaneously initiated in a preferred direction (Fig-2III-A,B,C). In this process of solidification: i) A narrow free melt surface (free melt surface never be zero) forms spherical shape under reduced gravity, therefore the melt experience weightlessness. Hence the hydrostatic pressure ( $P_H = \rho_I gh$ ) decreases [1,24,25]. ii) Initially, the argon gas filled in the conical region is entrapped, this argon gas occupy space below spherical shape, then gas pressure pushes away the tiny melt before freezing with the narrow free melt surface. Therefore solid is contactless in ampoule wall (self detached) and acts as the seed. iii) At this juncture triad of triple-phase line (TPL) forms spontaneously similar reported in [12], growth angle ( $\alpha$ ) will depend on its gap variation. iv) The freezing of melt procees further on this seed under same condition forming a concave meniscus shape with the wall of ampoule, and concave shape of crystal-melt interface (seen from melt top) Fig-2IIIA. iv) A gap is forms by vapor pressure in between ingot and ampoule wall and continues till the end of the growth. v) In detached growth, hydrostatic pressure decreases, it balances spontaneously, self-pressure difference built up into gap by an argon gas pressure. Detached growth in conical region is very vital as thermal stress, defect density, and surface cracks are naturally eliminated in the tapered end, and these are not propagated into melt. Only probability of vertical twin growth at the centre of ingot cannot be eliminated, it propagates continuously into melt.

In Fig-2IIIB, two variations: i) growth proceeds upward, then meniscus convert concave to straight, and ii) straight to initial stage of convex shape as reported in [16]. The interface convexity decreases as the hydrostatic pressure decrease with increase in ingot length, thus the pressure into gap increases by the argon gas plus degassing from the source materials, and it adjust itself the gap pressure variation. In Fig-2IIIC, if hydrostatic pressure is reduced to zero then meniscus has convex shape. At the end of growth, if concentration difference exists in melt and ingot, then melt forms spherical shape (convex) under reduced gravity (Renold number large). As grown InSb:Bi ingot showed Fig-2IV, and InSb:N attached Fig-2V. In addition, Fig-2IV showed a blackish band surrounded at ingot end growth (cap) for all the detached ingots. In attached growth observations, Fig-2V shows rough surface and unshaped end, where ampoule was not sealed with argon gas. Thus attached growth showed the presence of the gravity (Rayleigh) therefore in such cases, the ampoule had to be broken to remove the ingot.

The detached growths exhibited three observations: i) ampoule inner wall is covered with slight blackish thin oxidation layer in GaSb growth, ii) off white thin layer on ampoule inner wall in InSb growth, iii) for GaSb:Mn very clean inner ampoule wall. The oxygen might be the cause for first two observations. These features also seen in InSb:Bi and GaSb:Se growth. However, the industrial and commercial argon gas showed the scum on melt, a thin



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oxide layer and carbon inside ampoule wall, which degraded the surface smoothness and created defects in some detached ingots. Oxide layer can stick to the ampoule wall, form grain, twin and precipitate (Fig-3). A stacked layer (attached) below cap of the InSb:Bi ingot is seen. The results showed that the pure argon with low thermal vapor pressure variation is mandatory to obtain detached growth on earth [8]. *Detached growth in VDS confirms: without seed, without contact with ampoule wall, without coating, and without applied external pressures. It reveals that the VDS process act as the self-seeded, self-detached, self-controlled pressure difference and self-purification process [48-49, 51-53].* 

#### **III. 3 GAP VARIATIONS IN DETACHED GROWTH**

The three gap variations has been investigated (Fig-5) as: i) the gap remains constant, ii) the gap increases, and iii) the gap decreases. The process of gap formation could be decided by the triad formed by triple-phase line (TPL) of three phases: liquid (L), solid (S), gaseous (G)). However it is based on geometrical concept of meniscus [2]. Let  $\Theta$  be the contact angle,  $\alpha$  be the growth angle,  $\psi$  be the angle between two vectors into melt (LG and LS),  $\varepsilon$  be the angle between opposite vector into melt (SG) and adjunct vector along crystal-melt interface (LS) [52, 53]. Gap variation depends on the thermal contact angle ( $\theta$ ) and growth angle ( $\alpha$ ). Case I) if  $\varepsilon = 90^{0}$  and  $\Psi = 90^{0} + \alpha = \text{constant}$ , crystal grows with a constant diameter from a narrow melt free surface (optimum value), then the gap remains constant with growth (InSb: 172µm, GaSb: 128µm). Case ii) if  $\varepsilon < 90^{0}$ , then  $\alpha$  varies from  $0^{0}$  to  $30^{0}$  and crystal grows with decrease in diameter, and increase in gap (InSbBi:65-239µm) [40], therefore the melt free surface is just above optimum value and decreases with growth, Case iii) if  $\varepsilon > 90^{0}$ , then  $\alpha$  varies from  $30^{0}$  to  $0^{0}$  and crystal grows with increase in diameter, and decrease in gap (GaSbSe:154-45µm) [48,53], therefore melt free surface is just below the optimum value and increase with growth.

#### **III.4 PRINCIPLE OF THE DETACHED GROWTH BY VDS TECHNIQUE**

Based on the narrow free melt surface, we propose the detached growth mechanism in VDS. "Detached growth occurs in terrestrial lab, if a critical (narrow) free melt surface exists in ampoule, a tiny melt is pushed away from the wall forming spherical shape under reduced thermocapillary-buoyancy convection. A contactless growth has appearance in presence of pure inert gas at the optimum conditions and parameters. It is necessary to follow the growth cyclogram in suitable steps during the crystal growth as well as cooling of the crystal. Reduction in crystal diameter/ a gap formation reveals the reduced gravity may be comparable to the space grown crystals"

Crystal → Position	$InSb_{1-x}Bi_x x=0.02 x10^{-4}\Omega.cm$	$InSb_{1-x}Bi_x x = 0.06 x 10^{-3}\Omega.cm$	$InSb_{1-x}Bi_x x$ = 0.07 x10 <sup>-4</sup> \Omega.cm	$InSb_{1-x}Bi_x x = 0.08 x 10^{-4}\Omega.cm$	$InSb_{1-x}N_x  x = 0.005  x 10^{-4}\Omega.cm$	$InSb_{1-x}N_x x = 0.01 x 10^{-4}\Omega.cm$	InSb Undoped -3 x10 Ω.cm
1	5.52	1.02	1.42	5.04	5.35	4.27	3.11
2	5.45	1.00	1.47	5.08	5.52	4.34	2.99
3	5.45	1.02	1.51	4.71	4.93	4.49	3.31
4	5.55	1.02	1.56	4.84	4.91	4.61	3.24
5	5.48	1.06	1.49	5.00	5.48	4.51	2.94
6	5.51	1.04	1.53	484	5.21	4.25	3.18
Average $\rho(x)$	5.49	1.02	1.49	5.05	5.24	4.42	3.15
Energy gap eV	0.157	0.155	0.152	0.150	0.158	0.155	0.17

**Table-1** Dopants distribution into substrates of InSb: Bi and InSb:N bulk crystals, and the composition forGaSb:Seand GaSb:Mn is in similar order for crystal grown by VDS



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#### **III.5 ELECTRO-PHYSICAL MEASUREMENT**

Goal for these measurements is to investigate the effect of the detached growth on the composition distribution. Hall measurements by van der Pauw for InSb (mobility ~61k cm<sup>2</sup>/V.s), and GaSb (mobility ~1.2k cm<sup>2</sup>/V.s) were determined. The results of local resistivity VDS grown crystal confirm the high quality level of grown ingots. Infrared spectroscopy reveals low carbon and oxygen concentration as reported in [14]. We observed the radial composition variation < 5% for InSb:Bi and < 10% for InSb:N grown crystals. Similar results are obtained for GaSb:Se and GaSb:Mn crystals. The axial and radial variation is insignificantly observed above ingot shoulder, but not into the cone region. The radial composition is invariant for steady-state in regions above conical part. Experimental data contribute diffusion dominant growth by VDS process. It has resemblance to the  $\mu$ g lavel, and it is in agreement with numerical simulation in [24]. The uniform impurity distribution was obtained by forced convection on the ampoule rotating in the clockwise and anti-clockwise see Table-1. The increase in sensitivity of the substrates from the detached grown ingots is due to a shift in the donor – acceptor ratio with changes in the heat and mass transfer during growth process. The variation in the resistivity  $\rho$  (x) because of the charge carrier shift, while the high uniformity of the p (x) distribution is monitored with microuniformity.

#### **III.6 MORPHOLOGY OF GROWN CRYSTALS**

#### **III.6.1 MICROSTRUCTURE IN ATTACHED INGOTS**

Etched substrates from two different attached ingots are shown in Fig-3a,b. The substrates were cut from ingots body above conical part showed the twin growth from periphery of the substrates. The sticking of ingot to the crucible wall is associated with an increase of the dislocation density near the edge of the substrate, surface features shown as dislocation shapes in Fig-3c and Fig-3d respectively. At the edge Fig-2V, melt as well as solid in contact with the wall of ampoule, increased the thermal stress. The stress effect increases not only in the vicinity of the periphery, but also in the interior of the substrates. Etch pit density (EPD) at the edge of the wafer was order ~  $10^5$ cm<sup>-2</sup>. Pronounced dislocations including clustering slip lines and bundle-like patterns are observed on the surface of attached substrates. Measured EPD (> ~  $10^5$ cm<sup>-2</sup>) is higher than that of the mean value of average detached crystal being about ~ $10^{-3}$ cm<sup>-2</sup>. This correlation between the dislocation density and the hydrostatic pressure has influence on attached growth. The dislocations formed locally at the edge, rapidly spread across micro and macro distances by glide and multiplication processes as effect of inhomogeneties in the crystal.



Fig-5 SIMS graphs showing the bonding of the InSb:N and InSb:Bi detached ingots.



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#### **III.6.2 MICROSTRUCTURE IN DETACHED INGOTS**

The vertically cut substrates from region above the conical part of the ingot were mirror polished, then etched were used to determine EPD. In Fig-3e, f,g, h, etched substrate from four o different detached ingots are shown. The twin growth at the centre of the substrate in Fig-3g, and defect free substrates in Fig-3f, h, are seen without growth morphology. Etch pits are not detected near the edge of substrate cut from the detached ingots, where as the dislocation densities in the detached grown regions are of much lower order  $\sim 10^3$  cm<sup>-2</sup> and in some ingots EPD of the order  $< 10^3$  cm<sup>-2</sup>, i.e. two orders less than the attached ingots. Interestingly, presence of curved dislocations and the higher density near 1–2 mm at the periphery of the substrate was observed (Fig-6e). Reasons may be thermocapillary convection during growth as well as the influence of the surface tension analogous to space experiment of GaSb:Bi crystal [15]. Another reason may be, for the large free surface of melt, the melt surface convection move along the crystal-melt interface. In the presence of large free melt surface, Marangoni convection causes a non-uniform dopant distribution in the growing ingots [30]. The intensity of thermogravitational convection is reduced, and then thermocapillary convection effect increases by the presence of a large free melt surface. Under reduced gravity for quality and homogeneous crystals, it is mandatory to reduce the free melt surface to optimum value and lower radial temperature gradients. Reason for the microstructure growth is small temperature difference and surface tension gradient along the melt-gas (L-G) interface, which cause the melt flow (Marangoni convection). In presence of gravity, buoyancy and thermocapillary forces causes thermocapillary-buoyancy convection, which makes more complex growths [29,30]. Low defect density was predicted for near absence of hydrostatic pressure. The absence of the wall contact minimizes stresses in growth and transfer of stresses during cooling of ingot.

Items / Crystal →	InSb	InSb:Bi	InSb:N	GaSb	GaSb:Se	GaSb:Mn
Mobility( $\mu$ ) cm <sup>2</sup> / V.sec	$6.05 \text{x} 10^4$	4.45x10 <sup>4</sup>	$8.4 \times 10^3$	1120	3500	2500
Carrier Concen ( cm <sup>-3</sup> )	3.8x10 <sup>16</sup>	$2.58 \times 10^{17}$	$6.8 \times 10^{17}$	$2x10^{17}$	8x10 <sup>17</sup>	6x10 <sup>18</sup>
Resistivity (Ohm- cm)	3.0x10 <sup>-3</sup>	5.45x10 <sup>-3</sup>	0.5x10 <sup>-3</sup>	4x10 <sup>-3</sup>	5x10 <sup>-3</sup>	2x10 <sup>-3</sup>
Hall effect R <sub>H</sub> cm <sup>3</sup> /Coulo	-175	-24.5	-11.68	5.57	-17.1	-5.44
Micro-Hardness (H <sub>v</sub> )GPa	2.2	1.92	1.85	4.42	3.98	3.85
EPD $(cm^{-2}) \times 10^3$	0.96	1.2	4.1	1.1	3.2	4.9
Orientation	220	220	220	220	220	220
FWHM (arcsec)	65	87	115	97	124	145
Type of wafer	complex	n	n	р	n	n

Table-2 The typical structural and electronic properties of detached crystal grown by VDS

*Note:* Total crystal grown (since 1993) **72**, out of this InSb and doped InSb = **35**, GaSb and doped GaSb = **37**, out of 72 crystals, 85% detached, 15% entrapped into tapered end and 5% attached (see our reference).

#### III.7 EBSD

Electron backscatter diffraction (EBSD) in Fig-4 is conducted on the InSb:Bi substrate. EBSD forms different planes, diffract different electrons to form Kikuchi bands, which correspond to each of the lattice diffracting planes contains imaging of a section of typical analysis of sample. Grains and twins were not seen in orientation color map, that identifies the orientation of the crystallographic direction normal to the plane of the



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substrate surface. In Fig-4, a) Intensity quotient (IQ) map, b) color code map for orientation and c) inverse pole figure are shown. The scan colored region is  $115x265\mu m^2$ ; large area of EBSD mapping in orange color reveals the high crystalline quality of the detached ingot and it has advantage for crystallographic identity. No twin or grain growth morphology is seen in this map. A substrate cut transverse to the growth direction showed preferential [220] direction. In addition, this is confirmed by XRD and Laue backscattering. The map is useful for grain orientation, and grain boundary identification.

#### III.8 SIMS

Fig.5 shows SIMS analysis for InSb:N substrate. The strong presences of In-N bonds indicate successful incorporation of nitrogen. Number of Sb-N bonds is negligible as compared to the In-N bonds. Higher intensity of 'Sb' as compared to 'In' is due to relative sensitivity differences in the elements. In and Sb are in equal quantity as per EDS analysis. The presence of N-Sb bonds in the compound is negligible as compared to the number of In-N bonds. Free atomic nitrogen was not detected in SIMS of any of the three crystals of InSb:N. Fig.5 shows SIMS analysis for InSb:Bi substrate from the detached ingots. The strong presences of In-Bi bonds indicate successful incorporation of bismuth in compound. Number of Sb-Bi bonds is negligible as compared to In-Bi bonds.

#### **III.9 CHARACTERIZATIONS OF THE DETACHED INGOTS**

Characterizations such as SEM and EDAX, X-ray diffraction, Raman, FTIR, Vickers microhardness test and p-n junction devices are analyzed and published elsewhere [38-54]. The typical structural and electronic properties of InSb:Bi and InSb:N detached ingots showed in Table-2. The detached ingots grown by VDS have shown enhanced crystal properties, which confirmed in the VDS experiments. The investigation of entire detached crystal in terrestrial lab uncovers its mystery, and it is comparable to partial detached crystal growths in space.

#### **IV. CONCLUSION**

Detached crystal grown by VDS technique reveals that, the growth conducted in typical seven steps reduces thermal stress. The stable detached growth is achieved by - i) Inert gas pressure into ampoule, ii) Precise temperaturecontroller, iii) Design of ampoule, iv) Temperature gradients, v) Steady meniscus and interface, vi) Seven steps growth.

Detached growth conditions are: i) large wetting angle, ii) large growth angle, and iii) small gas pressure difference.

In all the grown ingots, the melt solidified at last showed the convex shape. An oxide layer is responsible for the scum formation and degrading ingots surface smoothness. EPD in detached growth is two orders lower than that of the attached, and it is comparable to a space grown crystal.

Detached growth has been confirmed by i) reduction in a free melt surface ii) optimum radial and axial temperature gradient, iii) vertical axial symmetry for ampoule position, iii) mandatory pure argon pressure into ampoule, and iv) precise control on growth condition and parameters.

The charge carrier composition distributions have attributed that the concentrations of the donors (Nd) and acceptors (Na) are close to carrier compensation.

The EBSD confirms the orientation and defect free single crystal growths, while SIMS analysis attributed to the uniform distribution of dopants and showed the bond formation.

Detached growth in VDS corroborates - without seed, without contact with ampoule wall, without coating, and without applied external pressures. It reveals that the process act as a self-seeded, self-detached, self-controlled pressure difference and self-purification.

Detached growth showed that the improved electro-physical and optoelectronic properties, enhanced structural and crystallinity, uniform dopant distribution at the macro and micro-homogeneity, and enhancement in crystals quality as compared to the crystals grown ever.

We have proposed a potential mechanism for the growth of detached crystal based on our experimental growths conclusion under terrestrial laboratory and technology. Our efforts to extend the process of detachment over the entire crystals are successful. The VDS has potential as commercial method to fulfill recent requirement of the quality grade substrates.



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