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The detached phenomenon and the fundamental science behind: The novel vertical directional solidification growth of the detached crystals by slow freezing

D. B. Gadkari*
Freelance Research & Consultant ; Crystal Growth and Technology
Embee-10A, Saibaba Nagar, Borivali (W), Mumbai-400092 India
Corresponding Author: D. B. Gadkari

ABSTRACT
The vertical directional solidification (VDS) process was developed to grow the Sb-based extremely high order quality crystals underground condition since 1993. The experimental results of detached growths are reviewed with focus on the growth of Sb-based semiconductors. The entire detached crystal by VDS-process in vacuum sealed ampoule by the prerequisite conditions of the crystallization by slow freezing of melt near crystal-melt interface is accomplished. The necessary conditions for crystalline solidification, the influence of different convections on high order crystal quality is investigated. The entire detached growths experimental data is analyzed. Based on investigation and analysis of the entire detached growth results by VDS-process, "the detached phenomenon and fundamental science" is reported.
Keywords: Solidification by freezing melt; Entire detached growth; III-V materials; Reduced thermo-gravitational effect

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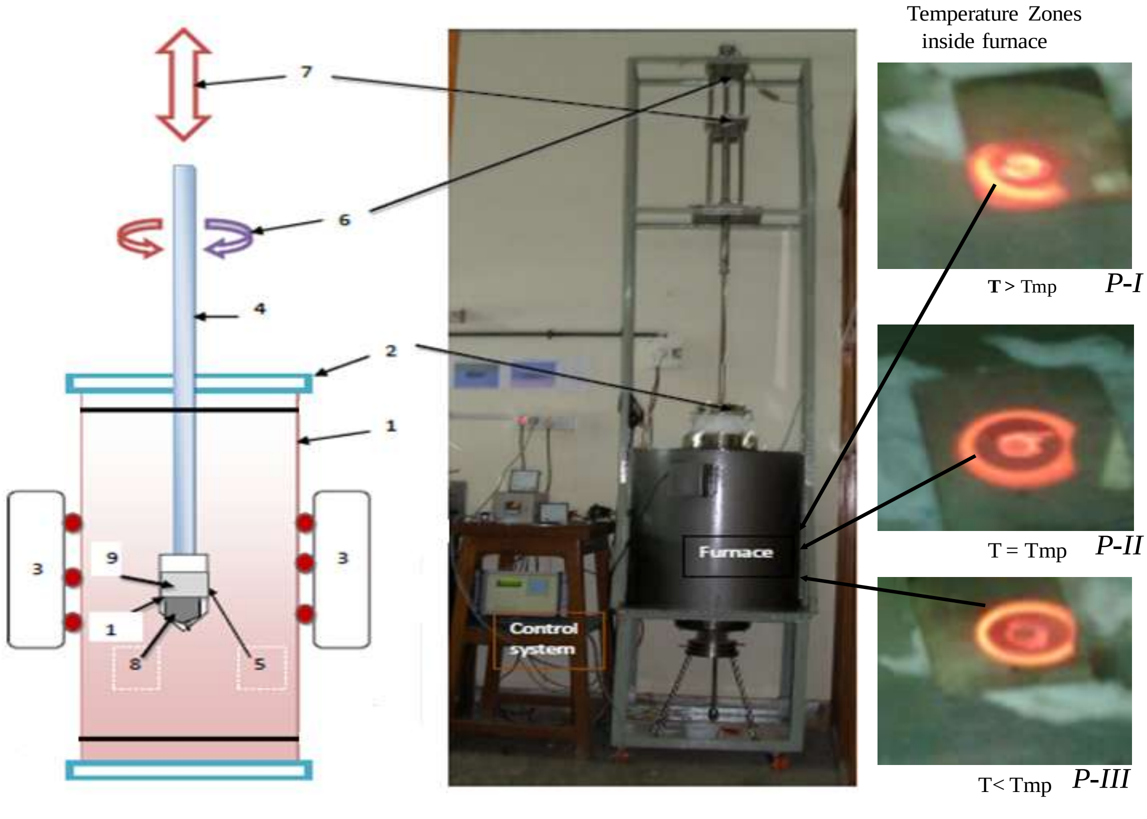
I. INTRODUCTION

Detached growth was found half century ago (1974), till date fundamental science is a mystery. The crystallization study in space is reviewed; the history of problem is considered. The influence of the crystal growth in space (microgravity or high vacuum) is investigated. The experimental data on partial detached growth in zero gravity is analyzed [1]. InGaSb growth is difficult underground condition due to the large gap of binary InSb–GaSb solid-liquid phases [2]. The gravity affects the convection in a melt and the heat and mass transfer [3]. In space, no atmosphere and pressure, so the dislocation density decreases in contactless crystal with a crucible, it increases for the crystal in contact with a crucible [4]. The binary and ternary alloy of Sb-based substrates are useful by modification of the band gap and the lattice constant by composition [5]. The experiments have been carried out to grow the high quality InGaSb crystals for application in electronic products; crystal is not available commercially by the constraint of InGaSb growth [6]. The concentration gradient of InGaSb depends on orientation at crystal-melt interface, diffusion mass transport (DMT) and heat mass transport (HMT) at interface front. Conductivity close to charge carrier compensation enhances crystallinity. The sensitivity of donor- acceptor ratio increases in shift change of DMT and HMT by for the reduction in thermo-gravitational convection. A quality of spreading resistance (Rs) corresponds uniform distribution, the homogeneous growth rate to the diffusion condition, which is predicted for the crystal growths underground and in space [7]. Microgravity environment improves crystal properties, and in depth understanding of influence by gravity on crystal growth needed [8]. GaSb:In crystals showed deteriorating properties, whereas InSb:Ga crystal non-deteriorating properties. The thermoelectric (TE) energy conversion is forecasted for the conversion of electricity [9]. The study of Ga-doped Ge crystal without crucible contact for the stable detached growth was reported. Experimental results show the structural perfection of detached crystal by the reduced thermal and thermo-mechanical stresses in contactless growth. The attached growths show surface striations and local defects, not yet fully clarified [10]. Next generation Sb-based ultra high-quality chips will require in Automotive, Medical, Artificial Intelligence, Avionics technology, and high speed digital communication in a tiny solid-state laser, flat-panel displays, radiation detectors, optoelectronics, and new generation lasers [11].

This research is focused on the detached phenomenon and the prerequisite conditions for the high order crystals quality growth by VDS-process underground condition. The gravity is hurdle in perfect crystals growth and the heat and mass transfer near crystal-melt interface. Necessary to understand problem of the gravity effect on crystal growth in a vacuum sealed ampoule. The problems of perturbing the homogeneous melt near interface front, and prohibiting the heat and mass transfer condition is investigated. The partial detached growth concepts in space [12-25], and the entire detached growth by VDS-process [26-44] are studied. The confirmation of the "Detached phenomenon and the fundamental science" is reported in ten-sections.

The entire detached crystals growth by VDS-process to study the detached phenomenon



a) b) c)

Figure- 1a. The schematic diagram of VDS process (CGRP-I), numbers -1) A quartz growth chamber (L=100cm, dia=10cm), 2) Wilson seal at both ends of quartz tube, 3) A vertical furnace with the thermocouples (red solid circles), 4) A vertical shaft to hold the ampoule into the centre of furnace, 5) A vacuum sealed quartz ampoule, one ends conical, 6) A clockwise and anti-clockwise rotation for mixing melt, 7) The up and down movement of ampoule for growth, 8) The detached ingot inside ampoule, 9) The vacuum (open space) above a melt, 10) The vacuum sealed quartz ampoule with source materials inside [33,35]. **1ba.** The photograph of VDS-process, the bottom Wilson seal is removed and the tripod used to hold the vertical quartz tube as growth chamber (CGRP-II). The outer circular ring is the quartz growth chamber, small inner circular ring is the quartz ampoule with melt. The ampoule moves downwards, the circular ring changes color : PI: the high temperature of melt $T>T_{mp}$ (inner small bright spot), PII: the initiation of seed growth $T=T_{mp}$ (inner small radish spot), PIII: the solid ingot's initiation of a seed growth $T<T_{mp}$ (inner small black spot).

II. EXPERIMENTAL PROCEDURE

The experimental VDS-process details in [26-31,33-42]. A schematic experimental set up showed in Fig-1a,b. Crystallization process in the furnace is in Fig-2a,b as grown ingots in Fig-3a-g. Inset Fig-IV is a first crystal growth furnace used for the detached growth by VDS-process. The initial step was pressure control of a inert gas in an ampoule and temperature controlled vapour pressure in evacuated ampoule. An ampoule with the vacuum 10^{-5}torr(~$1.3x10^{-3}$Pa) was purged for an oxygen impurity alternately for 10 times by refilling pure argon gas. Finally, the ampoule with a pure argon gas 200-300torr (0.027-0.041MPa) was sealed, an ampoule positioned vertically in an exclusive arrangement. The ampoule design (P-1): the ampoule having the conical tip of the specific angles (~45^0 to~75^0) was placed into the hot zone of the vertical furnace. In VDS experiment, the cyclograms [27-29, 32] of the heat supplied by vertical furnace was used the seven steps temperature profile (P-2)- i) the congruent and the homogeneous melt of source materials raised in 3hr to 850^0C, kept 3 hours for mixing Fig-3, ii) the ampoule with melt was lowered downwards 50^0C above melting point of materials (m.p.InSb-525^0C, GaSb-712^0C) in 3 hours by the lowering rate 10mm/hr, iii) to execute growth, the steady isotherm and steady thermal state maintained for homogeneous and chemically uniform melt for 3hr, iv) the sensitive growth process, an ampoule containing homogeneous and congruent melt was translated downward by slow freezing. The growth rate was ≤5mm/h (≤1.38µm/s). In this case growth time vary with ingots

The extremely high order quality crystals of Sb-based semiconductors grown by the entire detached bulk crystals using VDS-process underground condition

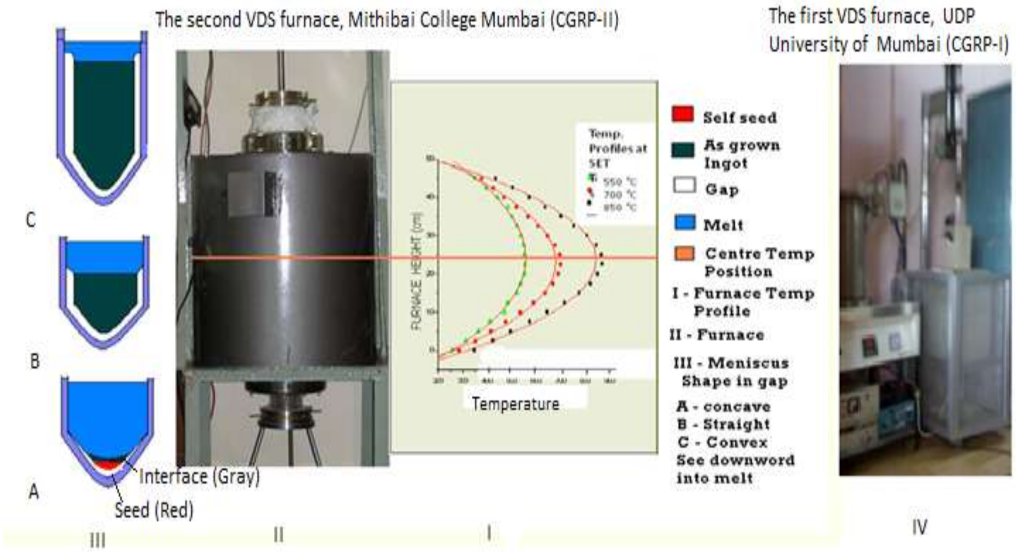


Figure 2, I) The stationaty vertical furnace temperature profile and the coloured indexed main part of VDS-process (the second VDS furnace- CGRP-II, 2007-2014). The temperature profile of the furnace for set temperatures (550^0C, 700^0C, 850^0C), II) The photo of stationary vertical furnace, was used for crystal growth, III) The spontenous self-seeded growth (red colour). The conversation of meniscus and interface at the different position of the growth (A, B, C). The meniscus shape for different positio: A-concave, B-straight and C- concex (seen from the melt). The melt top with large vacuum (the large free surface (blue coloured straight line). In VDS-process, the orange line in furnace centre shows growth three position $T_H>T_{mp}$, $T=T_{mp}$ and $T_{Cry}<T_{mp}$. Meniscus and interface steady and stationary in vertical furnace, ampoule moves downward opposite to growth direction and gravitational acceleration. Inset IV, the first very simple detached growth in vertical furnace (The first VDS furnace- CGRP-I, 1993-2006).

length (~ 4hr), v) an ampoule with grown ingot lowered 10mm/hr in 3hr below 100^0C of melting point (m.p.), kept at this temperature for the another 3hr for thermal stabilization of ingot, vi) the growth completion step, the furnace set temperature was lowered to 300^0C, then switched off for natural cooling for 2hr, vii) switch-off the furnace and wait for 1hr to cool grown ingot and remove ingot at the ambient temperature. The ingot growth total time ~30hr, the steps are explained details in [26-29,32].

Growth was performed by moving the ampoule downward relative to the stationary vertical furnace. The vertical growing crystal direction in a vacuum sealed ampoule is opposite to the direction of gravitational force or acceleration. The sealed ampoule with melt was rotated clockwise or anti-clockwise as forced convection. It was stirred to homogenize melt for uniform composition distribution into melt before crystallize of the to form crystal-melt interface forefront. An ampoule rotation probably promotes reduction in weight of melt column. The excessive heat of conduction is prevent by no downward support. Initial grown detached ingot act as a seed.

III. RESULTS

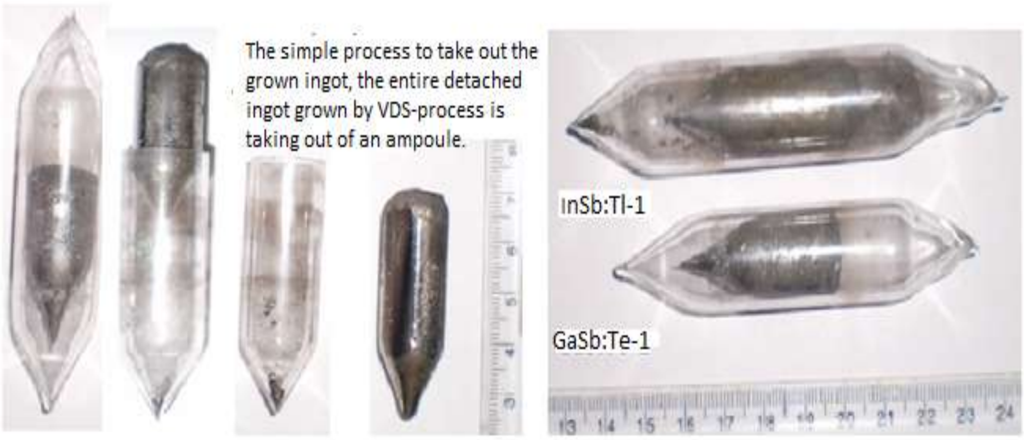
Crystallization by VDS-process, the study is divided into three parts- Crystal Growth Research Projects- CGRP-I, initial 1993 to 2006 [26-32], CGRP-II, 2007-2014 [33-39], and CGRP-III, 2014 onward [40-44]. The part- I and II, the entire detached crystal growth properties of extremely high order quality crystals grown by VDS-process underground condition were published. The dissemination of the analysis of property of the extremely high order quality crystals by VDS experiments was implemented. The part-III, the study is focused on the influence of gravity on the convections into melt, and effect on the properties of Sb-based crystals. The influence of gravity on a meniscus and the crystal-melt interface front to be investigated. To high light the "New Science and Technology" for the entire detached crystal growth and seek the conclusions of the partial detached growth in space for confirmation.

3.1 The preliminary features of the grown ingots

Detached or contactless Sb-based ingots came out easily from an ampoule by gently tapping on table, Fig-3c-f, Fig-4a-d. The single crystals with diameters 10 to 22mm and lengths L =55 to 85 mm in the preferred (220) crystallographic direction without a seed were grown. Ingots showed shinning and smooth surfaces, no micro-cracks, no defects, no striations, and end growth with a convex shape (cap). The featureless crystal proclaims

The ultra high quality crystals grown by the VDS-process underground condition

a) b)



c)

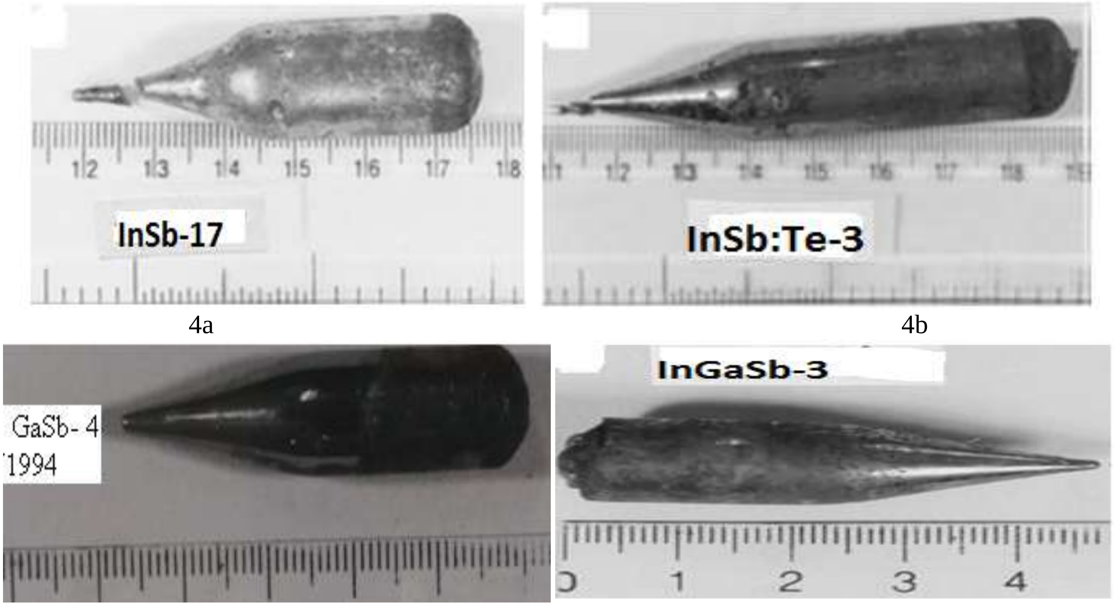
d) e) f) g)

Figure-3 The crystal grown in CGRP-II- a) as grown InSb-18 detached ingot into sealed ampoule, b) the GaSb-3 ingot's free movement inside the sealed ampoule, c) InSbTe-3, the initial small portion of ingot entrapped inside tapered shape, GaSb:Mn-3 the free movement of detached ingots, d) InSb:Bi-6, the free movements of detached ingot, e) the removing process of detached ingot by opening the one end of ampoule, f) the entrapped portion in conical shape of empty ampoule, g) the as grown featureless detached ingot removed out of the ampoule.

crystalline structure of solidification by slow freezing, and the preferred orientation growth from homogeneous melt near crystal-melt interface front. Grown crystals show flawless, no striations, no surface defects. The glossy surface appearance proves a single crystal growth. Crystals diameter were smaller than the diameters of an ampoule for contactless growth, ingots surface in Fig-3,4,8. These profound properties confirm significant control on growth parameters and conditions by the assessment of it. The spontaneous entire detached crystals grown by VDS-process. The three ingot growths (P-3) [37-38] were investigated- i) 80% ingots slide out easily from an ampoule, Fig-3e,g, Fig-4a-d, ii) 15% ingots entrapped into conical region, Fig-3e,g, and iii) 5% ingots attached to the

inner wall of an ampoule. Schematically the narrow gap formation showed in Fig-5a,b. The five growth governing parameters(P-4) are i) the thermo-capillary convection (Surface tension effect) ii) the thermo-capillarity concentration convection flows (Marangoni effect), iii) the increase in intensity of cohesive energy, iv) the decreased intensity of adhesive energy, v) a vacuum sealed ampoule [26-30,33-42]. The entire detached growth, the three conversions of meniscus shape (P-5) are- i) a concave shape, ii) a straight line shape, iii) a convex shape (cap) [41]. The conversion showed the concave Fig-2IIIA, the concave to straight line, Fig-2IIIB, the straight line to convex shape at the end Fig-2IIIC, Fig-3,4,5a. Schematic thermo-capillary influence showed in Fig-5b. A variation in meniscus and interface shape is confirmed along the detached growth axis [39]. The temperature difference was maintained into furnace, however, a self-pressure difference built up into the gap depends on the three gas requirements (P-6): i) a vacuum sealed ampoule, ii) the back filled pure argon gas, iii) the degassing from source materials. All detached ingots showed a convex shape at end growth that proves the influence of gravitational force is decreased in contactless growth. However, a few detached ingot growth showed the spherical solid drop at end growth Fig-8b [26-30,33-35,38]. The intense variations in convexity, and 'h-value' attributes as in Eqn-3 [33,39] two detached growth properties (P-7) - i) the lowest value of 'h' relate to the highest crystals quality, ii) the highest value of 'h' relate to the lower crystals quality. It is the evidence of detached growth. First time, the three gap variations (P-8) [39-40] are investigated: i) a constant gap (169μm), ii) a gap increases (20-145μm) [34], iii) a gap decreases (220-95μm [35]. The five

The ultra high quality crystals grown by the VDS-process In CGRP-I



4a 4b

4c 4 d

Figure-4 a) the detached InSb-16 ingot out of the ampoule, the homogrneous single crystal (3-D mass block) grown solidification by slow freezing in 1993 and b) InSb:Te-3 [26,30], c) the entire detached ingots growth, GaSb-4 1994 [30,33] and GaInSb-3 detached ingot grown in 2001[30,39].

growth cases (P-9) [41] were investigated- i) the inner wall of ampoule covers with slight blackish thin oxide layer Fig-3a (InSb-18), ii) off-white thin layer on ampoule inner wall in Fig-3c, iii) the detached ingots showed convex shape [26-31], iv) a few ingots have solid drop [29], v) the clean inner wall of an ampoule, Fig-3b (GaSb-3), the case-v strengthens the crystalline structure. The gap width depends[30] on the five self controlled process (P-10): i) self-seeded, ii) self-detached, iii) self-pressure difference, iv) self-defects free process and v) self-purification. The five fundamental parameters (P-11) of growth [26,32] are- i) Young contact angle in melt, ii) the critical growth angle, iii) the ampoule translation rate, iv) Rotation rate, and v) the set temperature. The vertical growth chamber at the steady isotherm, and thermal state showed the six basic melt conditions (P-12) [34-42] : i) the surface tension effect ii) Marangoni effect, iii) the thermo-capillary convection (meniscus), iv) the cohesive energy, iv) the concentration

convection flows into melt, v) the buoyant thermo-gravitational convection, vi) the steady external power supply by PID controller.

3.3 The basic of detached growth

In InSb detached growth, Indium (In) and Antimony (Sb) atoms (In-read, Sb-green, solid circles) at atomic level establish strong bonds by thermo-capillarity convection and the cohesive energy in presence of critically controlled surface tension effect and Marangoni effect, Fig-6a. In a vertical furnace a strong bond forms by the In, Sb atoms and InSb molecules under the optimum growth conditions and parameters. A tiny melt drop of homogeneous melt emerges 3-D structure of a melt by bonding energy. The entire dangling melt drop detaches into tapered end of an ampoule due to the binding energy Fig-6b. The crystalline growth set in motion serendipitously as the apparition of a gap due to solid growth from the dangling melt near crystal-melt interface front by the slow growth rate of solidification Fig-2. The homogeneous and chemically uniform structure of melt freezes near interface, hence mass block (single crystal) grow. The naturally, the preferential spontaneous growth select (220) direction of the crystalline structure under significantly decreased the buoyant thermo-gravitational convection into melt in a vacuum sealed ampoule. The solid ingot (single crystal) grows in temperature zone $T<T_{mp}$ Fig-1cPI-PIII. It acts as a seed as showed in Fig-1cPII (red dot at the centre) and Fig-2III-A (red color of concave curve). Detached crystal length continuously increases vertically with time, Fig-1cPIII (centre: small black dot as a seed), Fig-3c-f, schematically Fig-2IIIC. Melt forms concave meniscus shape with the inner wall of ampoule with grown crystal, i.e. blue curve shape Fig-5,6a [35,41]. The concave meniscus and concave crystal- melt interface is seen from melt, Fig-2IIIA,B,C. The initial thermal conductivity has large aspect ratio (melt

The schematic diagram of entire detached ingot by VDS-process underground condition

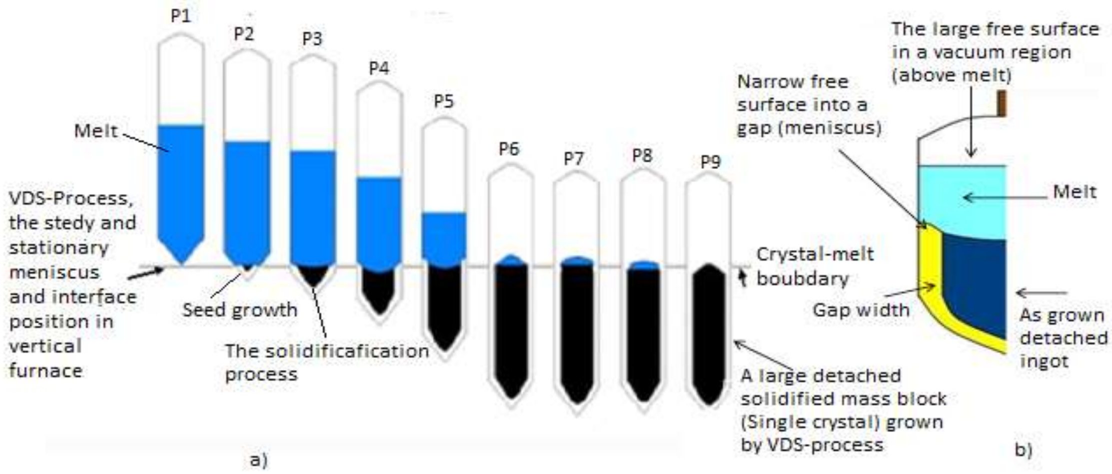


Figure 5 Schematic initial detached growth to till end growth [35-37], a) the steps involved solidification by slow freezing of melt at different stage by VDS-process for the detached growth of crystals (P1-P9). b) The capillarity, a gap (yellow color) in between the inner wall of ampoule (outer black line) and detached crystal (dark). The convex curve line is a narrow free surface (meniscus) above yellow color. The crystal-melt interface convex shape (crystal: dark and melt: sky blue), a large free surface (melt) in vacuum (white color).

height/crystal length), the shape has higher concavity as in Fig-5a and Fig-6a [30]. Detached crystal length increases, then the thermal conductivity of crystal increases, thus aspect ratio decreases, hence interface concavity decreases. The meniscus shape has conversion as the concave–plane–convex. The shape changes is seen in all entire detached crystals [30,39]. The crystal-melt-gas phases spontaneously generate triple-point-line (TPL) triad at interface, it continues up to end growth [39-40].

3.4 The problematic InGaSb growth

InGaSb growth is enormously challengeable, it is also complicated growth underground conditions, particularly for x=0.5 composition. InGaSb growth was not grown due to the difficulty cited in literatures for underground conditions. Hence, InGaSb crystal growth is cryptic. Because the large binary InSb-GaSb phase tempearture difference, it is ~100^0C between the liquidus (~650^0C) and solidus (~550^0C) temperature profile, is hindrance, particularly composition x=0.5 [2]. The novel vertical furnace in CGRP-II was designed to overcome the temperature difference > ~100^0C of the top and bottom temperatures of growth chamber (furnace) Fig-2I-III [39-40]. $In_{(1-X)}Ga_XSb$ (x= 0 to

1), twenty-two crystals growth of various composition were grown. The breakthrough and pragmatic growth results of the InGaSb difficulty, particular x=0.5, have been determined by VDS-process underground condition [33-44]. Grown InGaSb ingots showed shiny surfaces, no micro-crack, no dislocations, striations of the detached crystals Fig-8a,b. The physical properties showed in Table-1 [34-35,37-38,41]. The crystal characterizations were distinct and different properties. The peculiar case of InGaSb was the solid drop on ingot top grow by slow freezing due to the surface tension, the cohesive energy, the thermo-capillarity and concentration difference in a melt at the growing stage Fig-8b [28,30]. The crystalline properties have been studied by Hall measurement [28]. InGaSb detached crystal quality deteriorate with increase in composition of the Ga element Fig-6b [28,39], Hall measurements showed in Table-1 [34-35,37-38,41], it is investigated. The carrier conversions of n-type to p-type along the length of InGaSb crystal axis was measured by Hall method from the initial (InSb) to final (GaSb) growth showed in Fig-6b [28,39]. The carrier fluctuation explores near conversion point, i.e. n-type to p-type at middle of InGaSb ingots due to the charge carriers compensation. The new observation in a few detached growth that the crystals freezes without breaking of an ampoule up to 90% melt. The reason is that the ampoule do not cracks due to the expanding force on growing crystal longitudinal along the axis of growth direction as the expansion of InGaSb solid crystal is accommodated into melt. However, an ampoule breaks at the residual 10% melt, then scatters it. The cause is the expansion force of growing crystal transverse to the axis of growth direction, it is directed towards the inner wall of ampoule. Thus, the thermal stress is on the inner wall of ampoule as crystal undergoes differential thermal dilatation of diameter. The semiconductors properties is expansion on solidification by slow freezing. Thus, an ampoule breaks or cracks in attached or reattached growth to inner wall of an ampoule by the thermal stress. As result, the meniscusand a gap collapses, and no further solidification, thus InGaSb growth is incomplete. The basic mechanism of detached growths shows innovative science by slow freezing of melt [28,39]. The high order

The schematic diagram of a seed growth from dangling melt drop by VDS-process

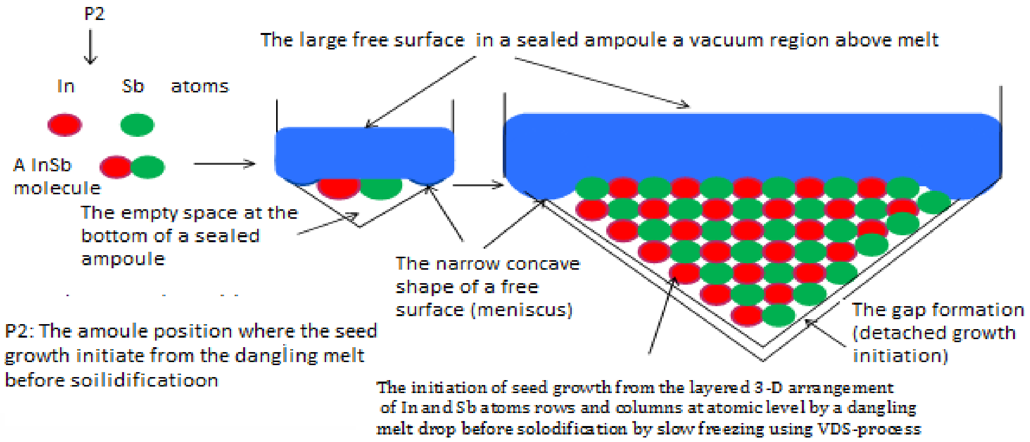


Figure-6a, The free atoms into homogeneous melt near interface (In, and Sb) initiation seed growth (P2 in Fig-a), The atoms and molecules, the ampoule moves downwards, then tiny melt solidify by slow freezing for Tcry < m.p. in a decreasing temperature gradient into stationary vertical furnace. In detached growth, only free crystal growth occurs by the self-organization of individual atoms by fulfilling prerequisite conditions at atomic level. The chemically uniform and homogeneous detached crystal grows by VDS-process.

crystal quality of detached $In_{0.5}Ga_{0.5}Sb$ crystal is evidence of the "Detached phenomenon" and basic science, it confirms.

3.5 Detached growth mechanism

The furnace temperature profile is essentially one of the fundamental characteristic behind the entire detached growth mechanism. The temperature zones in Fig-1, the constant high temperature at $T>T_{m.p}$ zone, melting point zone $T=T_{m.p}$ and the lower side crystal growth $T<T_{m.p}$ zone. In VDS-process crystal growth, the motion of atoms into homogeneous melt in a vacuum sealed ampoule resemblance the experiments cited in [45-48] to test effect of the falling bodies in a vacuum underground conditions. The aforesaid concept can be useful for the mechanism of fundamental science and the detached phenomenon by VDS-process. The basic mechanism of a seed growth in crystal growth zone, the homogeneous melt bulge away from the inner wall of an ampoule. It pulled inside by the increased strength of cohesive energy and thermo-capillary convection. The tiny melt's drop dangles into tapered end of ampoule, thus it solidify by slow freezing, and detaches unexpectedly by forming a gap (eqn-1, 2). Schematically the meniscus formation is shown in Fig-5aP-3, and Fig-6a. In presence of surface tension, the large enough Young's thermal contact angle on the ampoule wall and growth angles ensures radical increase in thermo-capillarity simultaneously by decreasing the buoyant thermo-gravitational convection into melt in a vacuum sealed ampoule. Thus unexpectedly by slow solidification (freezing) develops the entire detached growth process in a smooth quartz ampoule. All detached ingots form

convex shape (cap) of varying height "h" Eqn-3, this shape is proof of the entire detached growth by the complex mechanism [28,39]. Sb-based detached crystals properties by characterization showed extremely high-order crystal quality compared to the crystal grown by conventional methods in terrestrial lab. VDS-process corroborates the potential elevation of structural perfection [34-44]. In view of abovementioned explanation, the smooth entire detached growth by VDS experiments contribute the eleven points (P1-12) [26-44], are confirmed and these points divides in two category. The six conditions- 1) seven steps furnace temperature profile, 2) three gas requirements, 3) three gap variations, 4) six melt conditions, 5) five self controlled process, 6) five cases of detached growth. Moreover, the six parameters- 1) five growth parameters, 2) an ampoule design, 3) four growth governing parameters, 4) three meniscus conversion, 5) three type of growth, 6) the convexity variation, are based on experimental results.

The experiments to test effect of the falling bodies in a vacuum underground conditions (on Earth) was completed in 2014 [45-48]. It showed matters came down together very slowly, as no influence of the gravitational force on falling bodies with a constant acceleration. This effect is yet mystery. It is either fake time of weak gravitational force or acceleration (gravity), can be used in VDS-process.

3.6 The fundamental science of detached growth

The crystal growth from a melt in a vacuum either in space or on Earth, the molecular interactions and interfacial phenomenon have no influence, thus the intensity of buoyant thermo-gravitational convection into melt decreases. However, simultaneously the intensity of surface tension and Marangoni effects increases, are

Hall-van der Pauw electro-physical measurements at 300K

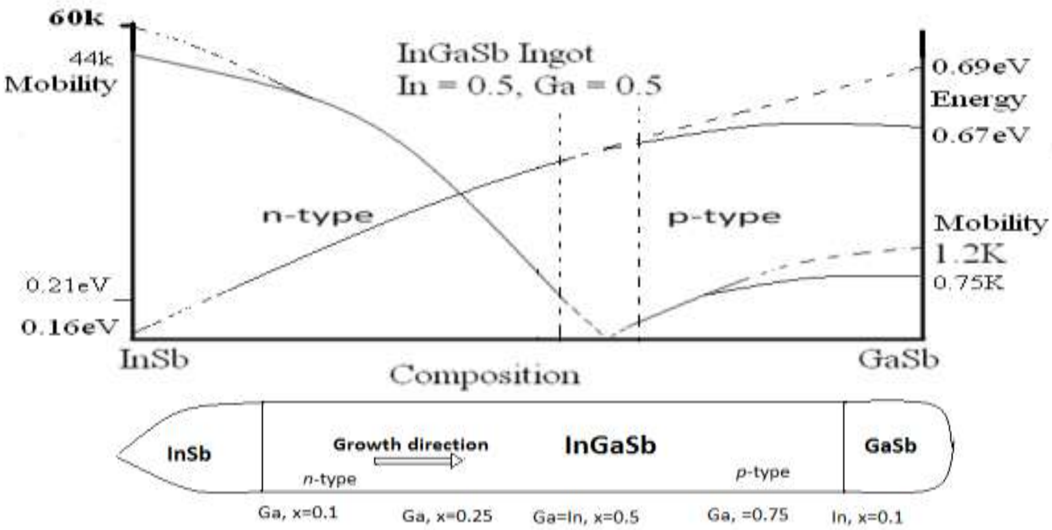


Figure-6b InGaSb (x=0.5) detached crystal showed the charge carrier conversation from initial growth n-type InaSb (conical shape region) to end p-type GaSb (convex shape region) along the vertical growth axis direction [33]. The energy increases and mobility decreases with increase in 'Ga' element in n-type region, to x=0.5 total compensation of charge carrier hence large flu-action. The energy and mobility increases with increase in 'Ga' element in p-type region up to x=0.75, later the energy and mobility decreases up to end growth with increase in Ga and decrease in 'In' element in the detached growth by VDS-process.

dominant into melt [1]. The experiment underground condition conclusion [45-48] is that the mass, pressure and density are independent of the gravitational force as well as the gravitational acceleration (in vacuum). In view of this, we propose that no effect of the hydrostatic pressure, the static pressure, and weight of the melt column on meniscus (gap) in VDS-process. Gravitational force/acceleration has no impact in a vacuum. The overall force balance has no effect on a meniscus as well as on a gap, because the furnace temperature is steady and stationary. The height of the melt reduce with increasing ingot length with time by changes in the aspect ratio. Study reveal- i) the controlled thermo-capillarity convection and the thermo-

capillary concentration convection into melt, ii) the slow freezing of materials at atomic level ensures only diffusion, iii) the doping distribution is practically uniform. Because the striations, composition variation, the inhomogeneity by changes in chemically structure into melt as well as in grown detached crystals were annihilated. Hence the high order of the detached growth crystals confirm- a) the buoyant thermo-gravitational convection, and the thermo-capillary concentration convection flows decreased by factor 10^2-10^3 order similar as reported in [20,21], b) the micro and macroscopic high order crystal quality, the homogeneous growth and structurally chemical uniform of Sb-based crystals satisfy the prerequisite DMT and HMT conditions as in [1,7], while exceptionally, c) the chemically uniform distribution of composition and homogenous growth rate associate the DTC and HTC. These three cases showed the improved physical properties [34-44]. First time, "Detached phenomenon" is confirmed, and corroborated it underground conditions.

3.7 Characterizations of detached crystals

The entire detached crystal growth by VDS-process conclusions are briefed, while the experimental result research papers published elsewhere for [26-44].

3.7.1 Dislocation density (dd)

The high-quality properties corresponds to the dislocation density (dd) [36,39]. Study conducted by etch pit density (EPD) showed the dd~10^3cm^{-2} has two cases. First case, FWHM <50 arcsec associates the perfect crystal for dd<10^2cm^{-2}. Second case, FWHM ~100 arcsec relatively higher order of the crystallinity by increase in the hardness with increasing dd up to dd~10^3cm^{-2}. For dd > 10^3cm^{-2} the crystals quality deteriorate with increases in dd due to the defects, so it is defective crystals.

3.7.2 Material hardness:

The study of hardness property of entire detached bulk crystal indicate its strength depends on interaction between dislocations motion mechanism and defect density [34-37, 41], e.g. the grain boundaries, and secondary phases. The dislocation-dislocation interaction increases dislocation density due to the dislocation entanglement drags motion. The plastic deformation occurs because the simultaneous breakage of large number of bonds instead of dislocation motion The perfect binary crystal such as InSb, and GaSb crystals showed the extremely high strength (hardness) [36,39]. Nearly perfect single crystal with low dd <10^2cm^{-3}, the

Growth morphology in detached ingots grown by VDS

Fig-7a Scale: 500µm --- Fig-7b Fig-7c Fig-7d

Figure-7 Growth morphology of GaSb-4 detached crystal grown by VDS, a) for the growth of curved dislocations, the surface tension and thermo-capillary convection flows through meniscus inside the interface during the detached growth, b) the uniform and homogeneous vertical twin growth at the centre of the substrate in a few detached growth show the thermal stress during seed growth, c) the substrate surface shows no growth morphology due to the homogeneous steady and stationary melt by reduced buoyant thermo-gravitational convection, and controlled thermo-capillary concentration convection (Marangoni effect) near interface, d) The substrate conical region right side is edge of substrate (near dark region), it attributes the optimum surface tension and Marangoni effects [41].

micro-hardness of grown crystals were studied for the alloy and the doped crystal [33-41].

3.7.3 Micro-hardness measurements

The hardness measurements of detached crystal showed increase in hardness with a certain low value load, later it decreases with further increase in load. This variations in properties showed high order of micro-hardness [41]. The plastic deformation occurs because the simultaneous breakage of large number of bonds instead of dislocation motion. The perfect binary crystal such as InSb, and GaSb crystals showed the extremely high strength (hardness) [36,39]. The micro-hardness of grown crystals of the alloy and the doped crystal were studied [33-41]. Nearly perfect single crystal with low dd <10^2cm^{-3}, the hardness of detached crystal initially increases and then it decrease by increasing dopant [36,42] and alloy composition changes into melt in ternary crystals as showed in Table-1 [28,39]. Typical hardness is for InSb-16 Hv= 2.2GPa [29], GaSb-4 Hv = 4.42GPa [33].

3.7.4 X-Ray diffraction

XRD back scattering diffraction pattern of the entire detached crystal showed the crystal's high order crystallinity with the low FWHM, i.e. 72arcsec of InSb-16 [29,30,35,42], and 54arcsec of GaSb [33,34]. The dopant added [36,37] and ternary alloy crystal [38,41] showed increased values of FWHM due to defects formation. The internal crystal structure appear with the spots in the diffraction pattern. The detached crystals R-factor, the binary crystal higher value than ternary crystals. For GaSb-3, R-factor value is four. X-Ray diffraction pattern analysis was highest for crystalline binary binary- InSb, and GaSb [30,33]. The study of detached doped crystal growth- InSb:Bi, InSb:N, InSb:Te GaSb:In, GaSb:Se [39] confirms highest crystallinity

3.7.5 Raman Spectroscopy

The appearance of only TO phonon for InSb [33] and GaSb [30,33] by backscattering showed (110) growth direction. It confirms the single orientation of the detached growth by VDS-process underground condition, Raman scattering measurements are convenient and sensitive method for probing defects, additionally spectra shows the absent of defects [33-39]. Typical samples showed the anti-stokes Raman spectrum of zinblende structure [33-38].

3.7.6 FTIR Transmission

FTIR transmission graphs shows maximum transmission >35% for binary materials, the energy gap for GaSb-4 is Eg = 0.68 eV, and InSb-16, Eg = 0.16 eV. Energy gap close to the intrinsic energy gap to the perfect detached crystal growth quality respective crystals. The increases in dopants and alloy crystals correspondence to low transmission due to decrease in crystallinity by defects. **3.7.7**

Hall electro-physical measurement

Hall-van der Pauw measurements have been analyzed to discover the crystal quality of the detached crystals at 300K. Electrical measurements are shown in [33-42]. The physical property improvement for the stable state of the detached crystals (main body of crystal, after shoulder) showed decreased density of the dislocation. The crystal lattice mismatch of grown crystal ($\Delta a/a \leq 1\%$), and the decreased ratio of mobility variations ($\Delta\mu/\mu \leq 10\%$). The properties of binary crystals attribute the increase in micro and macroscopic homogeneity of the entire detached crystals. In VDS-process, the enhanced crystal properties of the detached growth corresponds to the steady and stable state of the interfaces as well as growth parameter and the conditions. The crystal growth process for high order crystals quality of the micro-homogeneity variation in resistivity, carrier concentration and mobility are caused due to the changes in heat and mass transfer

The InGaSb entire detached ingot by VDS-process underground condition

Fig-a) $In_{0.5}Ga_{0.5}Sb$, Fig-b) $In_{0.5}Ga_{0.5}Sb$

Figure-8 $In_{(1-x)}Ga_xSb$, x=0.5 detached crystal growth by VDS-process, a) the grown polished ingot's shiny surface reveals the missing surface micro-crack, no defects and striations, b) the grown ingot end growth shows the solid drop on top due to the surface tension and concentration difference in the solidification process. The initial 90% ingot growth in Fig-a was detached growth; the polished shinning surface showed perfect crystalline structure by detached growth. But ingot showed reattached to the inner wall of ampoule for residual 10% melt, and scattered due to cracks by the constitutional super cooling and the thermal dilatation of the growing ingot [29].

conditions at crystal-melt interface for the steady and stable position in vertical VDS furnace. The plane or near plane meniscus shape (slightly concave, or convex) and the decreased concave interface shape region showed the largest mobility, than crystal grown ever, or in commercial brochures. The carrier mobility smooth increase along the ingot growth axis shows the stationary conditions of growth and homogeneous distribution. It reveals increase of crystalline quality as compared to the doped and ternary alloy crystal. GaSb and InGaSb crystals properties highlighted in Table-1, composition variation in Fig-6b. The mobility(μ) is highest for detached InSbμ_n=5.9x10^{4}cm^{2}V-^{1}s-^{1}, GaSbμ_p=1060cm^{2}V-^{1}s-^{1} [35,36]. The mobility of crystal grown by VDS-process underground conditions is higher than crystals grown ever by the conventional methods. The resistivity (ρ) and spreading resistance (Rs) along the radial and axial direction showed uniform distribution. It correspondence to the homogeneous melt as well as grown detached crystals. The resistivity along diameter and growth axis confirms the high order of crystals. The carrier concentration very close to intrinsic concentration dni (InSb ni=2.1x10^{16}cm^3, and GaSb ni=1.7x10^{17}cm$^{3)}$, thus concentration ratio of donor (dn) or acceptor (da) with the intrinsic concentration (ni), i.e dn/ni or da/ni, close to unity. The sensitivity increases near unity ratio for high order quality crystal. Hence high crystallinity crystal's concentration shift changes in DMT and HMT, and the homogeneous growth rate specify DTC and HTC, is verified for detached growth by VDS-process in terrestrial lab. The crystal sensitivity proves by incident laser beam, photo-conductivity, thermoelectric heat effect, forecasted for electricity by thermoelectric (TE) energy conversion [42].

3.7.8 The n-type to p-type conversation InGaSb x=0.5

InGaSb (x=0.5) grown by VDS-process and studied by electro-physical Hall measurements at 300K [28,39]. For InGaSb middle crystal, the charge carrier conversation of n-type (InSb) to p-type (GaSb) along the growth axis increases sensitivity of the donor-acceptor ratio for compensated detached crystals. The shift changes by sensitivity of high order crystal quality is confirmed **3.7.9**

Devices fabricated

Device fabricated on substrate of the detached crystal at 300K, the p-n junction [43] and Schottky and MOS on InSb substrates structure [44] operates at the high [44]
at the high temperature 300K, it showed dark current<0.1nA.The conventional devices operating temperature was up to180K. Devices characterization with low dark current confirms high order quality of detached crystal [43,44].

3.7.10 EBSD and SIM

InSb:Bi and InSb:N doped crystal substrates thickness ~ 200μm were characterized. The composition variation was <1% for dopant distribution. InSb:Bi EBSD IQ mapping showed a large solidified mass block (single crystal) [42] is confirmed. The SIM of InSb:N showed incorporation 'N' atoms into InSb chain structure [42]. It corroborate micro and macroscopic homogeneity of the entire detached crystal superiority [37,38].

3.7.11 Growth morphology

In Fig-3,8, the featureless surfaces confirm the controlled convection of the thermo-capillarity and thermo-capillary concentration near interface front. In a vacuum, no effect of pressure and gravitational force or acceleration. Hence, the buoyant thermo-gravitational and the concentration convection flows were apparently decreased many fold into melt. The substrates cut from radial and axial direction of the entire detached crystals were studied to understand the slow freezing effect on the thermal mechanism and solidification. The EPD study of entire detached growth showed absent of growth morphology and microstructures on surface, however, some ingot's grow complete different features by VDS-process Fig-7a-d. In thermal mechanism and solidification process, the curved microstructures inside away from the edge of substrate was assigned to melt flows due to thermo-capillary and thermo-capillarity concentration convection flows Fig-7a,b, The convection near the crystal-melt interface depends on thermal condition by slow freezing [39]. It is tangential through the meniscus, which is directed towards interface [42]. The star feature grown at the centre due to eutectic temperature of Sb element and growth size decreases from centre towards the edge of substrate [30]. A uniform straight line lamellae at centre of substrate grows by thermal stress, and continue throughout uniform size vertically in detached growth [42]. The entire detached crystal growth (mass block or single crystal) properties are totally confirmed. The microscopic growth morphology by the slow freezing at atomic level ensures the heat and mass transfer condition only by diffusion. Detached growth experiments results conclusion since 1993 published elsewhere [26-44], the some specific publications are referred here: InSb [34], GaSb [35] GaInSb [38], InSbTl [36], InSb:Te [39], p-n junction iode [43], Schottky-MOS structures [44], and doped (Se, Bi, N, Mn, Mg etc.) [39].

3.8 Gap formation process

The gap is independent of pressure difference, but depends on the temperature difference in a vertical furnace as well as in a vacuum sealed ampoule of VDS-process. No static pressure, and the weight of melt column on meniscus as well as on the gap in the vacuum sealed ampoule. The physics of gap formation cited in literature, however the theoretically gap was evaluated from the geometrical concept and growth conditions: The case-I, a gap unstable, if θc + α < π, while in the case-II, a stable gap, if θc + α > π, showed in Fig-5,6. Simple mathematical derivation a gap thickness (e), however, the equations is cited in [15,21]

$$e = \frac{Ra}{\cos\theta c}(\cos\theta c + \cos\alpha) \quad (1)$$

$$e = \frac{2\gamma}{\Delta p}(\cos\theta c + \cos\alpha) \quad (2)$$

$$h = \frac{2\gamma\cos\theta c}{\rho g Ra} \quad (3)$$

Where θc: Young's thermal contact angle, α: a growth angle, e = R_a - R_c: the gap width between the ampoule radius (R_a), and the crystal radius (R_c), γ: the melt surface tension, Δ_T: the thermal temperature between top and bottom of vertical furnace or Δ_P: the pressure difference between an argon gas top of melt (P_H) and bottom(P_C) of an ampoule in gap. The meniscus height (h), the pressure (p) and density (ρ) is independent of gravitational force in a vacuum sealed ampoule.

The sealed ampoule height ' H' acts as a vacuum chamber in VDS-process. An ampoule of height H=0.1m used for the detached crystal growths in VDS-process, simple mathematical derivation for a vertical velocity (v) is cited in [45-48].

$$y = y_0 + v_y t \frac{1}{2} a_y t^2$$
(4)

$$0 = H - \frac{1}{2}gt^2 \quad (5)$$

$$H = \frac{1}{2}gt^2 \quad (6)$$

$$v = \sqrt{2gH} \quad (7)$$

$$v = \sqrt{2gH} = \sqrt{2x9.81x0.1} = \sqrt{1.96} = 1.4 m/s \quad (8)$$

Ampoule height 'H' is half field by melt in this growths, the fluid movement not given by eqn-8. Because the solidification by slow freezing is insignificant at the interfacial front cause is the surface tension gradient becomes zero. The rapid proliferation in viscosity, no effect of the convection, conduction and circulation at growing crystal at interface forefront underground conditions as similar in [45-48].

3.9 The entire detached growth by VDS-process

In VDS-process the entire detached growth study focused in CGRP-III, 2014 onward [40-44] to investigate the "New Science and Technology" behind it. The entire detached and high order crystal quality prime difficulty is the influence of gravity underground condition. The detached crystal growth and basic science is discussed. **3.9.1 Explore detached growth: in space and by VDS**

The experimental result conclusion is investigated, it is reported for the some partial detached growth in space (microgravity or high vacuum), and the entire detached crystals grown by VDS-process underground condition.

The moving meniscus model (MMM) [12,13] that the

Table-1 Detached InGaSb growths

The undoped and doped growth of binary and ternary alloy showed crystalline quality at T = 300K for samples not annealed. The mobility showed the complex (two type - n and p) charge carrier behavior [30]. Physical properties and electro-physical measurements showed highest crystalline structure for InGaSb detached growth [29].

Sr. No.	Crystal	InSb	InSb :Te-3	GaSb-4	GaSb:Te-2	GaSb:In-1 In = 0.5	GaSb:In-2 In = 0.25	GaSb:In-3 In = 0.15
1	In (gms)	9.68	9.72					
	Ga (gms)			20.95	6.48	2.92	4.76	3.61
	Sb (gms)	10.24	10.28	37.16	11.42	10.21	11.12	7.00
	Dopant: (gms)		0.35 x10^{-3}		0.38 x10^{-3}	4.82	2.62	0.66
2	Setting temperature	800	820	850	810	850	850	850

	(0^0c)							
3	Growth Temperature (0^0c)	575	575	762	762	762	762	762
4	Freezing rate (0^0C/ cm)	32	32	32	32	32	32	32
5	Up/Down Rate (mm/hr)	5	5	5	5	5	4	4
6	Rotational Speed (rpm)	10	10	10	10	10	10	10
7	Cone Angle (0^0)	54	57	62	67	72	65	74
8	Gap width (µm)	69	112	95	124	145	151	139
9	Crystal length (mm)	60mm	60mm	72	66	50	52	42
10	Crystal diameter (mm)	12mm	12mm	22	18	12	14	10
11	FWHM (arcsec)	65	87	49	98	128	112	95
12	Orientation (Laue,/Raman	220	220	220	220	220	220	220
13	Energy gap (Eg)(eV)	0.16	0.18	0.69	0.73	0.46	0.49	0.51
14	Mobility10^3 (cm^2 / Vcsec)	58.9	26.9	1.12	0.12	1.1	3.26	4.96
15	Resistivity 10^{-3} (Ohm-cm)	3.02	0.41	4.45	5.25	3.18	7.78	1.94
16	Hall effect (cm^3/ Coulomb)	-165	-10.6	5.57	-6.16	3.44	-7.21	- 7.06
17	Carrier Concen10^{17} (cm^{-3})	0.38	5.8	2.1	16..6	18.1	86.8	88.6
18	Argon pressure (torr)	275	280	260	278	242	250	275
19	Disloca density x10^3 (cm^{-2})	0.62	2.3	0.67	1.4	1.2	2.29	3.91
20	Micro-Hardness H_v (GPa)	2.25	2.1	4.42	4.12	3.72	3.85	3.98
21	Crystal Growth	Detached	Detached	Detached	detached	Detached	Detached	Detached
22	Semiconductor type	complex	n	p	n	complex	n	n

detached growth on earth was more difficult than in the spacecraft. In VDS-process, the entire detached growth was not difficult by the 'Steady and Stationary Meniscus' (SSM) in the vertical furnace. The detached crystal [14] accomplishes two factors - i) the coating interior of crucible wall to avoid oxidation, but the coating was difficult, ii) the buoyancy thermo-gravitational convection reduction was prerequisite condition. In VDS-process, these two factors were eliminated using a vacuum sealed ampoule and high purity argon gas, therefore, no coating for the solidification by slow freezing. Experimental set-up [15] was designed to grow InSb and GaSb crystals in spacecraft, the meniscus trouble relates to oxygen contamination. In a vacuum sealed ampoule in VDS setup, no oxygen contamination by high purity argon gas, proves spontenous increase in the growth angle, and the Young's thermal contact angle. However, the self-temperature difference in a growth chamber and in a gap (self-pressure difference) is predicted. It opens new area of Science and Technology on Earth. In space [16], the dewetting phenomenon confirms the influence on quality and growth mechanism, it reveals counter-balancing the hydrostatic pressure to benefit the increasing structural quality. But in VDS the meniscus shape depends on the gap by intensity of the cohesive energy and optimum control on the surface tension, and thermo-capillary. Whereas the pressure, density and shape of melt were independent of the gravitational force in a vacuum sealed ampoule, as in [45-48]. Therefore, no static pressure and static weight on meniscus was existed at the reduced buoyant thermo-gravitational convection, it benefits by increasing thermal angles and structural quality. GaSb dewetted growth [17] was without coating and the low purity back filling inert gas, but InSb showed attached growth. Because the chemical interactions at the interface by dissolute oxygen disturbs a contact angle. So, the low purity argon gas is required for the detached growth experiments in space. All these difficulties of crystals grown in space were self controlled and eliminated by VDS, as replied above for the references [12-16]. Dewetting phenomenon [18] in the space confirms that - i) it is not related to pressure differences, ii) the growth rate and rotation rate is independent, iii) the crystal structural quality enhanced in space crystal than terrestrial lab, iv) it also opens the new research area for the New Science and Technology in space, as well on Earth. These conclusion of crystal grown in space and conclusions of entire detached growth by VDS-process underground condition are in good agreement as replied in references [12-16]. In fact, the ampoule containing melt slowly translated downward in the direction of gravity in steady and stationary VDS vertical furnace however, the crystal growth is vertical direction and it is opposite to the gravitational force, it benefits for the entire detached growth. The dewetting phenomenon [19], the freezing process confirms a meniscus slides smoothly on the crucible wall for a stable dewetting, it showed reduction in defects in spacecraft, than the terrestrial lab. Therefore, the dewetting hypothesis in space totally confirms. The partial detached growth in the space showed high perfection of these crystal regions. In VDS-process the similar reply is given in [12-16]. The entire detached crystals by VDS-process underground condition is ideally high order crystal quality, i.e. perfect crystals showed alike the partial crystals grown in space. Therefore, the detached phenomenon and fundamental science behind the crystals grown by VDS-process underground condition is totally confirmed, also replied in [12-16]. The partial detached growth in space [20-21], the crystal grown away from the inner wall of crucible then the defects related to the contact of melt were eliminated. In space [22], the thermo-capillary convection and surface tension were reasons for curve dislocation growth. Theoretical study in [23-25], first self-consistent thermo-capillary model was reported the heat transfer narrow gap (meniscus). Gravitational effect of static pressure and buoyancy-driven flows are challenges for detached growth on earth. Melt static weight on meniscus disrupts force balance, the static pressure changes with time affects a gap. The concepts are indistinguishable and in good agreement with the VDS-process experiments results underground conditions.

3.9.2 The parameters corroborate VDS-process

The crystal growth experiments underground condition are difficult due to the buoyant thermo-gravitational convection. The growth requires the medium vacuum to decrease the gravitational force, influence of the thermo-capillary convection (surface tension effect) and the thermo-capillary concentration convection (Marangoni effect) flows into melt, which inhibit DMT and HMT process. The prerequisite conditions in VDS-process underground condition is need to control the influence of convections into melt near interface. Because, the free crystal growth occurs by self-organization of individual atoms, and molecules at atomic level by only diffusion. However, these problems have been eliminated in detached growth by VDS-process underground condition. The entire detached growths conclusions attributed to micro and macroscopic homogeneity of high order crystal quality, and showed homogeneous composition and chemically uniform structure. The crystal growth rate relate to the DTC and HTC at decreased buoyant thermo-gravitational convection. The experimental results by the entire detached growth by VDS-process showed the extremely high order crystal qualities and properties. The cause is that only diffusion transfer, the hypothesis is successful by VDS-process underground conditions. The conclusions are confirmed by the Hall electro-physical measurements. The physical properties and growth morphology showed different, and the lower dislocations density. Because, the buoyant thermo-gravitational convection decrease at the controlled optimum surface tension and Marangoni effect. Growth of the star like complex feature by eutectic growth (for excess Sb) is at centre and size reduction extending towards edge of the substrate but far away from edge. It showed dislocation density is larger at the centre than the near edge of substrate. The defects such as- stress, contamination, and heterogeneous nucleation are eliminated. The thermo-gravitational convection is reduced and Marangoni influence is decreased due to the mass transfer into melt near the crystal-melt interface by controlling only pure diffusion. The striations from inhomogeneity composition are eliminated / controlled, the point defects formation by the dopants are decreased. The entire detached crystals growth properties results make known the high order of perfect crystal (singe crystal) by VDS-process.

3.10 The crystal quality by the detached phenomenon

In a vacuum, the gravitational force decreases, then surface tension and Marangoni effects increases it affect detached growth and crystal quality. The latter influential forces is challenge to control into melt. However, the aforesaid forces have been controlled successful in VDS-process underground condition. The conclusions are specified in this article.

3.10.1 Specific hypothesis of entire detached growths

In detached crystal growth by VDS-process, the crystal dimensions, growth morphology, optical properties, and characterizations of optoelectronics device have been studied. The quality is estimated by measurements and characterization [26-44]. On the basis of ambitious interpretation of the entire detached crystal growth by VDS-process underground condition. The entire detached growth phenomenon and the fundamental/basic science behind is concluded on the VDS experimental results

The Sb-based detached crystals have extremely high order quality. Semiconductor melt convection has low viscosity and high surface tension at the elevated temperature. First and foremost requirement by VDS-process is medium vacuum underground condition. Use the PID controller to minimum temperature fluctuation by external effects. Determine the ampoule translation rate by thermo dynamical condition for the solidification rate necessary for slow freezing. VDS experiments, the axisymetric crystallizer axis to orient vertically to the rotating motion of ampoule to be maintained, and inclination of an ampoule to be avoided. The detached crystal properties depends on the angle of inclination with vertical direction of the ampoule and the crystallization rate. In VDS-process, the freezing rate is calculated from the thermodynamic of Sb-based crystals, and chosen 1/10 of calculated value to corresponds conditions to the diffusion and the heat and mass transfer underground condition. The argon pressure is large above the large free surface (melt) and very low in a gap below very narrow free surface (meniscus) in sealed ampoule for the optimum vacuum. The growth chamber (quartz tube), top end closed by Wilson seal and bottom end open to ambient temperature and temperature difference to be maintained. The thermo-capillarity convection into a gap near triad point decreases if narrow free melt surface (meniscus) forms by the capillarity. But the optimum radial temperature gradient and critically high axial temperature gradient in melt at the steady and stationary temperature profile of a vertical furnace are necessary.

Study of growth morphology of featureless crystal of the micro-surfaces for the entire detached crystals signify the uniform distribution radial and axial direction. The direction of the morphology flows with direction of the transverse convection flows into melt near crystal-melt interface is tangential through the meniscus. The uniform surface growth morphology without striation, and no segregation on the transverse cross sections of substrate is confirmed. The morphology grow away from the substrate edge inside, while larger dislocation density at the centre of the a few ingot is verified. The longitudinal crystal growth axis showed symmetric and uniform micro twin lamellae at center of crystals in vertical direction. The EPD showed lower dislocation density at the edge and relatively larger at the centre. The striations are absent, therefore, the entire detached growth (single crystals) is homogeneous, and chemical uniform structure at the macro and microscopic growth level. The inhomogeneity into melt significantly reduces in the radial and axial distribution into melt near interface by VDS-process, it increases crystallization. The macro and microscopic inhomogeneity is abruptly declined into the entire detached crystals underground conation. In VDS-process, the convective flows into melts corresponds to the homogeneity and structure of melt as well as the in grown crystals. The control on micro and macroscopic inhomogeneity of the high quality crystal grows by eliminating perturbing factors by gravity. A uniform composition and dopant distribution is obtained throughout the entire detached grown crystals (single crystal). The surface tension (thermo-capillary), and thermo-capillary concentration convection flows (Marangoni effect) are controlled, and a narrow free melt surface underground condition is achieved.

The entire detached crystal growth correlate to the parameters of crystallization process. The high quality detached crystal is very sensitive to the Marangoni effect in the reduced buoyant thermo-gravitational into melts underground condition, is confirmed by Hall van der Pauw electro-physical measurement. The VDS-process provide homogeneous melt inside the sealed ampoule, and need vertical axial symmetry for high quality crystal growth. The reduced buoyant thermal gravitational convection by gravity and control on the surface tension and Marangoni effect is achieved. Surface tension effect and Marangoni effect dominate in a large free surface in a very high vacuum, then it is very difficult to grow the entire detached crystals in space or on Earth. The latter creates convection flows into melt as obstacle for the detached growth, and the disturbances in uniform structure and the dopant distribution into the melt, thus in grown crystals. VDS-process experiments results of the entire detached growth discover the heat and mass transfer only by diffusion, which result exceptionally the high order entire detached crystal underground condition. The influence of convection increases the thermo-capillary concentration convection (Marangoni effect) needs to control, then the significant heat and mass transfer occurs. The vertical crystallization direction of grown crystal axis is opposite to the gravitational acceleration (gravity) in a vertical furnace. The increase in transfer rate correspondence to changes in thermo-chemical uniform structure at micro level. The changes in meniscus conversation from concave-plane-convex and the crystal–melt interface convexity decreases along the vertical direction is investigated [30,39]. The high order crystal by the critical growth parameters and conditions (P-1-11) is basic prerequisite in VDS-process and its interpretation contribute to the basic of growth process The entire detached crystal growth by VDS-process [26-44] is confirmed underground condition.

Experiments of detached growth is repeated process using VDS-process and has potential and bright future for the III-V an II-VI semiconductors growth. The study of entire detached crystal could discover the fundamental Science and Technology of semiconductors' crystal growth underground condition by VDS-process.

3.10.2 Newly proposed detached phenomenon

The first time, concise specific concepts of the entire detached crystal growth is defined as the detached phenomenon underground conditions. However, the concepts have been extracted from the experimental results of Sb-based crystal grown by VDS-process.

In VDS-process, the homogeneous melt was pulled inside away from the inner wall of vacuum sealed ampoule by thermo-capillarity and cohesive energy near the crystal-melt interface. As the buoyant thermo-gravitational convection is drastically reduced at the controlled (optimum value) the surface tension and Marangoni effect into homogeneous melt. Hence, the atoms and molecules into melt forms the homogeneous 3-D structure by bonding from homogeneous melt. The 3-D structure of a melt by binding energy dangles in the conical shape of an ampoule. Then subsequently an ampoule (with melt) translated downward by slow rate into the stationary vertical furnace at elevated temperature to lower temperature zone. A melt drop solidify by slow freezing near interface due to steady temperature difference of top and bottom of the furnace. The solidifying crystal serendipitously forms apparition of a gap conjure the detached crystal growth away from the inner wall of an ampoule forming very narrow free surface (meniscus) due to the temperature difference in a growth chamber and in an ampoule top and bottom (with melt). A temperature difference developed into the ampoule top of melt (vacuum) and in a gap. The pure argon gas and degassing from a melt enters into a gap; these gases occupy below the very narrow free surface, i.e. concave meniscus (seen from melt top). The three-phase lines (TPL) form a triad due to the Solid-Liquid-Gas phases near the concave crystal-melt interface. However, the crystalline structure solidify near interface by slow freezing the dangling homogeneous melt continually till the end growth (convex shape) with increase in length with time by reducing aspect ratio. The detached growth process is continuously established with the conversion of meniscus shapes concave-plane-convex, simultaneously by reducing the crystal-melt interface convexity along the crystal growth axis. The detached growth occurs without breaking of the solidification process or no perturbation into homogeneous melt. Because the surface tension effect (thermo-capillarity), and Marangoni effects (thermo-capillarity concentration convection) are controlled to the optimum value into melt. however, the buoyant thermo-gravitational convection and the convection flows into melt are drastically decreased. The entire detached crystal growth by VDS-process, the constant diameter, the increased diameter and the reduced diameter due to the gap by triad variation along the growth axis is investigated, it is totally confirmed. The entire detached crystals qualities are proved exceptionally high order crystal qualities by the characterization and crystal properties. In spite of this, only free crystal growth occurs by the self-organization of individual atoms and molecules at the atomic level, because of the fulfilling the prerequisite conditions growth. The free crystal grows only by diffusion is the innovative phenomenon by VDS-process underground condition. The high micro and macroscopic uniform properties of grown crystal, it is necessary to minimize the effect of the perturbations on the heat and mass transfer processes by reducing the thermo-gravitational convection during growth and minimizing surface tension and Marangoni effect to increases the stability of heat and mass transfer processes into melt. In VDS-process experiments are performed underground using well studied optimum growth parameters and conditions. The crystal growth axis direction is opposite to the gravitational force or acceleration. In view of these conclusions the "detached phenomenon" is totally confirmed by VDS-process underground condition.

IV. CONCLUSION

The entire detached crystals of the varying diameter and length grown in sealed ampoule by VDS-process underground conditions. Experimental results of VDS-process, a meniscus shape conversion, the reduction in interface convexity and variation of the gap is investigated. The exceptionally high order crystal quality of the entire detached crystals grown by only diffusion is verified. VDS-process fulfilled the DMT and HMT condition, is confirmed by the properties of grown entire detached crystals. The existence of the reduced buoyant thermo-gravitational convection, and control on thermo-capillary convection and thermo-capillary concentration convection flows have been proved, on the basis of physical properties, the growth morphology and the Hall measurements. Experimental results of detached crystals showed the prerequisite optimum of the basic growth points (P1-12) as the growth conditions by VDS-process. The macro and microscopic homogenous, and chemically uniform structure of the entire detached growth by VDS-process is higher than the crystal grown ever by the established technique and commercial broachers. The entire detached growth properties by VDS-process is alike conclusions for the partial detached crystals grown in space, is verified. From the experimental results and aforesaid conclusions, the detached phenomenon is totally confirmed by VDS-process underground condition. The basic science behind the detached phenomenon has been proved in a vacuum sealed ampoule underground condition. VDS is the new potential technique for the bulk growth of the reactive materials such as III-V and II-VI group semiconductors, and for the future development of the "New Science and Technology". In addition, applications are proposed in Electronic industry, Digital communication, and hard-to-predict spin-offs to new categories of electronic products, and the image courtesy hardware-Multilayer Switch Feature Card (MSFC).

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CV: ACADEMIC PROFILE:

Dr D B Gadkari, received B Sc (Physics) degree (1974) from Shivaji University, Kolhapur and M.Sc. degree in Physics from Bombay University (1976), University of Mumbai confirmed M Phil (1986), Ph. D. (1998) under UGC-FIP scheme. He joined Mithibai College Mumbai India (Nov-1976) as a Asst Prof, HOD- Physics (2000-14), Asso. Prof (2006-2014), Vice-Principal (2010-2013) and Principal-I/C (2013-2014). He was Adjunct Research Guide-Faculty of Science: University of Mumbai (2014-2017). Freelance research and Consultant: Crystal Growth and Crystal Technology since 2017. He has been published 55 articles in referred journals and 32 proceedings. The member of professional International and National research journals. He has successfully completed Research projects (nine) sponsored by Government of India, University of Mumbai, and parent institute (SVKM) Mumbai India. The new growth process using vertical directional solidification (VDS) was developed, and Indian patent (1994) is in his name. This concept has been opened new research area in New Science and

Technology by VDS-process. Received awards/medals/certificates from the academic and the research institutions / colleges in India and abroad.

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