Acta Astronautica Vol. 11, No. 3–4, pp. 155–162, 1984
Printed in Great Britain.

0094–5765/84 $3.00 + .00
Pergamon Press Ltd.

ON BASIC RESULTS OF THE JOINT SOVIET–VIETNAMESE PROGRAMME "HALONG" ON SPACE SEMICONDUCTOR TECHNOLOGY

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(*Received* 12 *September* 1983)

Abstract—The $Bi_2(Te,Se)_3$ and GaP crystals were obtained according to the joint Soviet–Vietnamese programme "HALONG" on semiconductor material science on board the "SALYUT-6" orbital station during the U.S.S.R.–S.R.V. international space flight.

The comparative investigations of these crystals and their Earth-grown analogs were carried out in order to reveal peculiarities of the heat and mass transfer process during crystallization under microgravity. The special attention was paid to the studying of growth morphology, bubble formation, impurity and composition distribution in these crystals.

The strong influence of the crystallization conditions on the impurity segregation mechanism and structural perfection of these materials were exhibited. It was also found that electrical–physical parameters are correlated with the crystalline structure and the composition distribution of crystals.

1. INTRODUCTION

In connection with perspective development of the space processing the investigation of peculiarities of crystallization process and behaviour of the multicomponent semiconductor compounds under gravity-free conditions would be of great importance. As it is known, many physical-chemical properties of growing semiconductor crystals depend strongly on the heat and mass transfer mechanism during crystallization process. The later is mainly determined by the hydrodynamic state of a liquid phase that is distinguished in space and on Earth. Therefore, comparative studies of space- and Earth-grown crystals can reveal the influence of gravity on crystallization process and its features under different growth conditions. According to this trend the joint Soviet–Vietnamese programme "HALONG" on the semiconductor material science was performed on board the SALYUT-6–SOYUZ orbital complex during the U.S.S.R.–S.R.V. international flight by the cosmonauts L. I. Popov, V. V. Gorbatko, V. V. Ryumin and Pham Tuan[1]. In these programmes six technological experiments were carried out. Two among them were dedicated to the growth of $Bi_2(Te,Se)_3$ solid solution crystals by the directional crystallization technique in the "Crystal" and "Splave" furnaces: the Halong-1 (HK-1) and Halong-6A (HS-6A) experiments, respectively. The Halong-4 (HK-4) and Halong-5 (HK-5) experiments were performed for purpose to obtain the undoped and (Zn,O)-doped GaP crystals, respectively, by the traveling solvent method (TSM) using the temperature field of the "Crystal" furnace. In this paper the main results of studying $Bi_2(Te,Se)_3$ and GaP crystals obtained in the Halong-1,4,5,6A experiments will be discussed.

2. EXPERIMENTAL METHODS

2.1 *Directional crystallization*

Directional crystallization of $Bi_2Te_{2.7}Se_{0.3}$ solid solutions was carried out with the maximum heating up to $T = 850°C$ and keeping at this temperature for a period of 120 min (thermal soak) to achieve thermal equilibrium in the system and homogenization of the melt. Then the cartridge was pulled out with a rate of 0.188 mm/min for HK-1 and by regulated cooling at a steady rate of 2.8°C/hr for HS-6A experiments. The synthesis procedure of initial materials and preparation of technological ampuls were described in paper[2]. Analogous experiments under similar thermal conditions were taken out on the Earth.

After the surface morphology investigation by SEM, the ingots were sectioned into halves along the growth axis. The distribution of $Bi_2(Te,Se)_3$ crystal composition was investigated in longitudinal and cross sections by electron microprobe analysis[2]. The local distribution of Seebeck coefficient was also studied. Besides that the transfer phenomena in the range of $2 < T < 300$ K and the Hall coefficient in the magnetic field up to 70 kOersted at Helium temperature were investigated on the samples cut from middle part of space and Earth-grown ingots[3].

2.2 *The* GaP *crystals were grown by the crystal furnace*

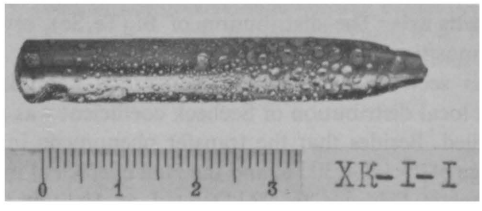
The LEC single crystals with a cylindrical form of 9-mm-dia. and 5 mm of length oriented in (111) direction were used as a seed. The liquid zone thickness was chosen of the order of 3–5 mm taking into account the residual acceleration on board the orbital station $g = 10^{-3}$–$10^{-4} g_0$. In order to avoid the possible drift of crystals and the change in the zone length due to leaking gallium special quartz fixing

rings were used. The gallium solution was not preliminarily saturated by phosphorus. It was suggested that during furnace heating the wetting with liquid gallium and partial dissolving of the seed and source GaP crystals took place that provides the good contact between the crystal-seed and the nutrient solution.

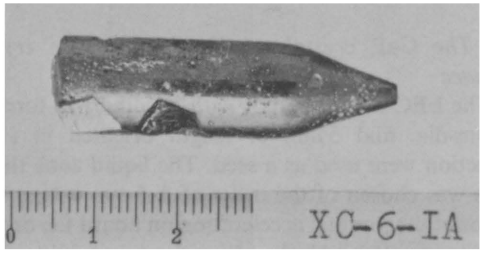
The undoped (experiment HK-4) and (Zn,O)-doped (experiment HK-5) GaP crystals were grown. The crystal growth took place in temperature range of 1000–1080°C with a temperature gradient in liquid zone of the order of 20–40°C/cm and 10–15 hr of duration.

The surface morphology and crystalline perfection of GaP crystals were studied by means of optical microscopy, X-ray diffraction topography and scanning electron microscopy. The crystal growth rates were determined by the division of the layer thickness by the growth duration. The Hall effect was studied by Van-der-Pauw method in order to determine carrier concentration and their mobility. Cathodoluminescence of GaP samples was measured with the standard method.

In order to determine quantitative parameters of the growth process the exact thermal conditions must be known. To study temperature profiles and dynamic characteristics of the "Crystal" furnace specialists from the U.S.S.R., the S.R.V. and the D.D.R. jointly developed the apparatus and carried out the "Imitator" preliminary experiment[1]. A temperature profile in the "Splav" furnace was calculated by computer solving heat conduction equation for the given geometrical configuration of this system taking into account thermal conductivity of the capsule and semiconductor ingots. Crystal growth rates for HK-1, and HS-6 experiments were found based on these results[2] and the phosphorus diffusion coefficients in liquid gallium were also calculated from the data of HK-4 and HK-5 experiments[4].



(a)



(b)

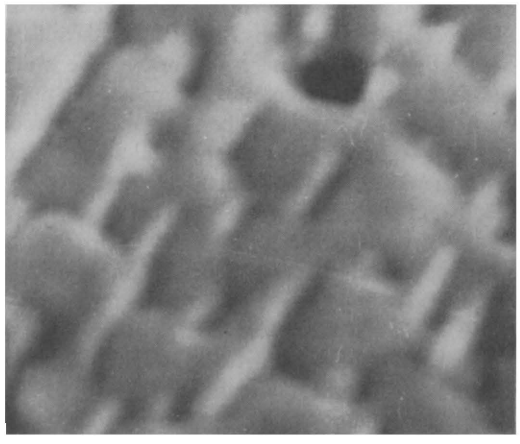
Fig. 1. (a) The space-grown ingots HK-1 and (b) HS-6.

3. SOME RESULTS AND DISCUSSION

3.1 $Bi_2Te_{2.7}Se_{2.7}Se_{0.3}$ *solid solutions*

3.1.1 *Surface morphology.* General feature of the space-grown ingots is the presence of different pores and large hollows on their surface (Fig. 1). Formation of large hollows and negative face, as it is shown in[5] is caused by non-steady-state growth conditions. Significant difference is observed while investigating the surface morphology of space- and Earth-grown crystals by SEM. The surface of space-grown ingots are characterized by well developed relief (Fig. 2a) that indicates the weak contact (wetting) of the melt with the confining quartz wall.

The surface morphology in the conic bottom of the space ingots HK-1 and HS-6A are shown in Fig. 3. As seen, the space samples are characterized by the presence of free surfaces where the transition from dendritic growth (at the initial region of ingots) to the directional growth of oriented crystals is observed. The formation of dendritic parts (Fig. 2b) is apparently caused by a high crystallization rate of the super-cooled melt[6]. While comparing the morphology of initial parts of space- and Earth-grown ingots it can be assumed that a considerably higher



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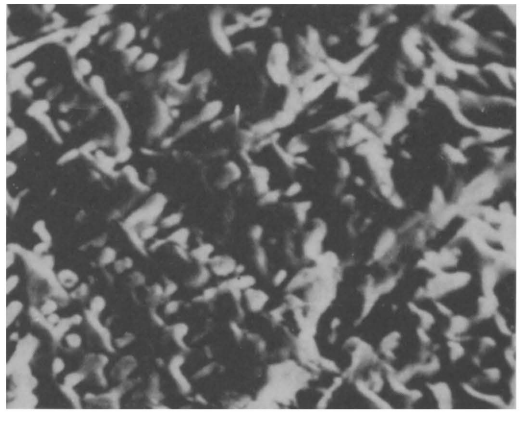
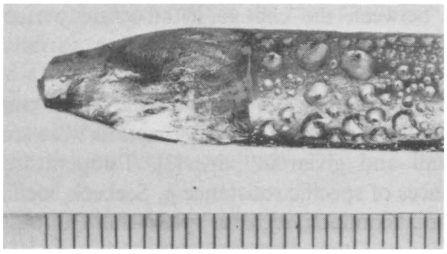


(b)

Fig. 2. (a) Wrinkled surface of the space ingots and (b) the dendritic areas in the initial part of these ingots.



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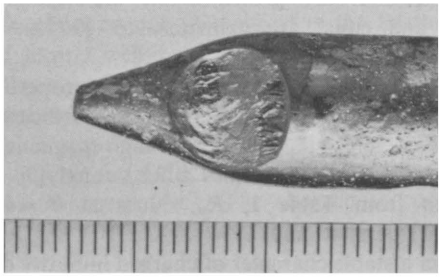
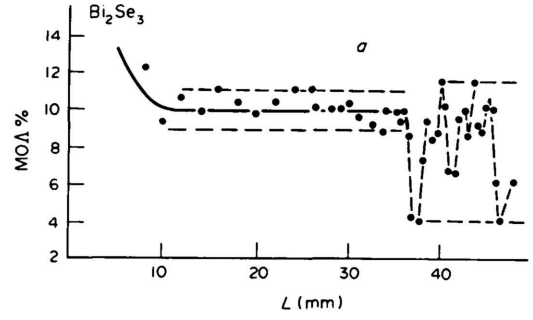


(b)

Fig. 3. (a) Surface of initial part of HK-1 and (b) HS-6 space-grown ingots.



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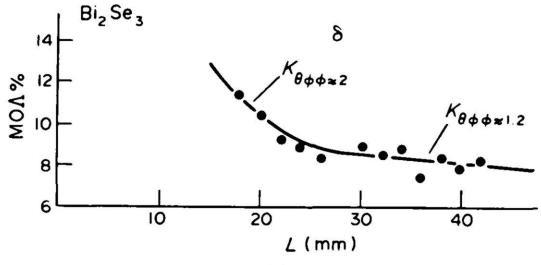


(b)

Fig. 4. (a) Bi_2Se_3 distribution along space and (b) Earth-grown HK-1 ingots.

supercooling of ΔT is possible for the crystallization in space than in the Earth conditions. The supercooling value, probably depends on the hydrodynamic state of the liquid phase affecting the process of formation of the critical size nuclei. It should be mentioned that experimental dependences of ΔT on the convection intensity during the crystallization from the solution were also observed in paper[7].

3.1.2 *Composition distribution.* The Bi_2Se_3 concentration profiles along the space- and Earth-grown ingots obtained in the "Crystal" furnace (HK-1) are illustrated in Figs. 4(a,b). As seen, the composition distribution is very different in these crystals. In case of the space-grown ingot the Bi_2Se_3 distribution in relatively homogeneous on the large distance with the selenium effective distribution coefficient $K_{eff} \approx 1$.† This indicated that the quasi-steady-state of diffusion-controlled segregation was achieved (Fig. 1a). In corresponding region of the Earth-grown crystal the Bi_2Se_3 content monotonously decreased (Fig. 4b) that follows from normal freezing equation[2]. The change in the Bi_2Se_3 distribution is occurred at the end of HK-1 space grown ingot (Fig. 4a). During the sharp increase of growth rate the composition fluctuations with large amplitude (from 4 to 12 mol% of Bi_2Se_3) and period about 4 mm are observed. The composition variation in this case correlates with the surface morphology peculiarities. There are different hollows at the corresponding part of crystal surface (Fig. 1a) which indicate the non-equilibrium nature of solidification process. In the Earth-grown ingot the composition variation is monotonous even after such sudden change in growth rate (Fig. 4b).

Analogous result is obtained for the experiment HS-6A, in whiles also there were fluctuations of growth rate. The Bi_2Se_3 distribution along space- and Earth-grown ingots is shown in Fig. 5. As seen, the composition of the HS-6A space-grown ingot is characterized by large macrofluctuation with decreasing period and amplitude. The clear correspon-

†The $C_s^{\infty} \approx 10$ mol% and the deviation C_s is about 1 mol%.

Table 1. Ratio of Hall coefficient measured in I, II, III cross sections and on face *a*, *b* for space (k) and Earth (*L*) samples

Sample No.	$\frac{R_I}{R_{III}}\Big\|_a$	$\frac{R_I}{R_{III}}\Big\|_b$	$\frac{R_b}{R_a}\Big\|_I$	$\frac{R_b}{R_a}\Big\|_{III}$
		$H = 20$ kOersted	$T = 4.2$ K	
HK-1-K	1.9	2.1	1.6	1.7
HK-1-L	1.5	0.6	0.7	1.7
		$H = 70$ kOersted,	$T = 1.6$ K	
HK-1-K	1.25	1.32	1.38	1.46
HK-1-L	1.44	0.65	0.72	1.58

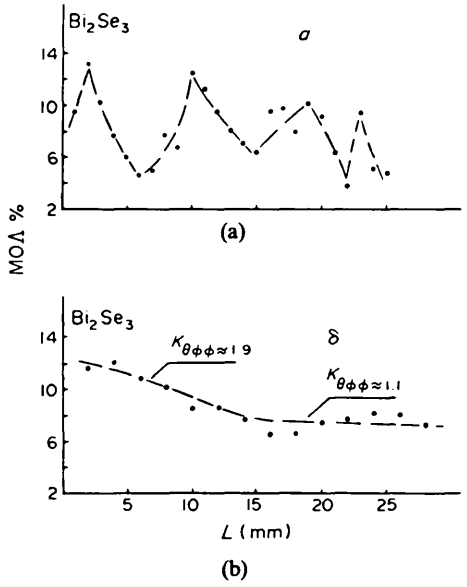


Fig. 5. (a) Bi_2Se_3 distribution along space and (b) Earth-grown HS-6 ingots.

dence between the change in structure perfection, surface morphology and composition variation is also observed[8].

3.1.3 *Electrical-physical properties.* The transfer phenomena in these $Bi_2Te_{2.7}Se_{0.3}$ crystals were studied in detail and given in paper[3]. Temperature dependences of specific resistance ρ, Seebeck coefficient α, thermal conductivity κ for space- and Earth-grown samples were illustrated in Fig. 6. Using the data of ρ, α and κ the thermoelectric figure of merit z of $Bi_2Te_{2.7}Se_{0.3}$ material was calculated. The value of z for space and Earth-grown samples are close to each other and equal approximately to $10^{-3}K^{-1}$ for $T = 100$ K (Fig. 6).

In spite of the similarity in integral properties of transfer processes, the value of Hall coefficient R_H and character of its variation in high magnetic field for space and earth samples differ strongly[3]. As it is seen from Table 1, R_H values at $T = 4.2$ K, $H = 20$ kOersted and $T = 1.6$ K, $H = 70$ kOersted confirm a stable character of charged impurity distribution along the HK-1 space sample whereas for

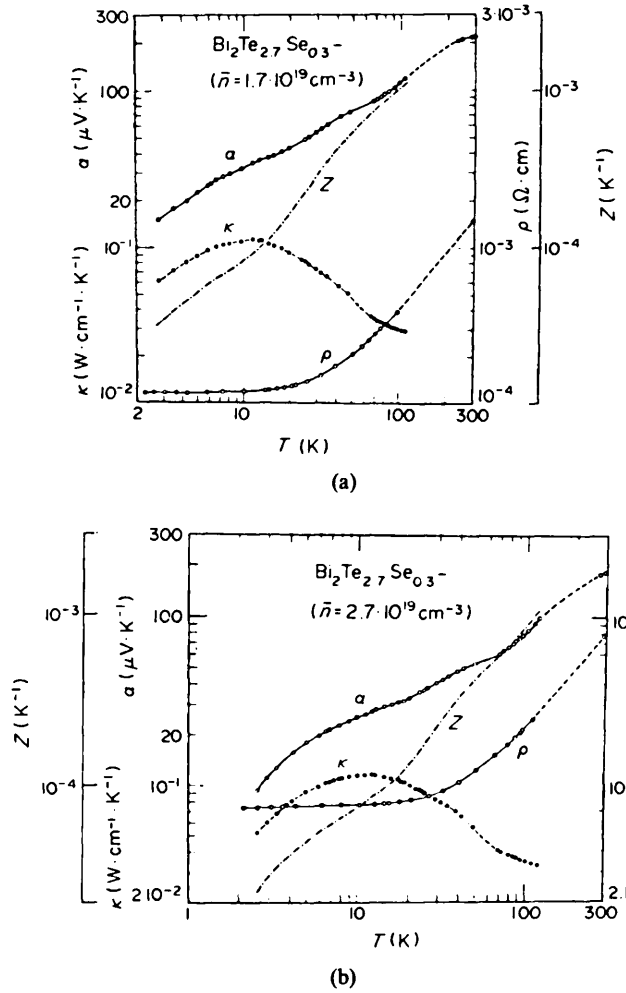
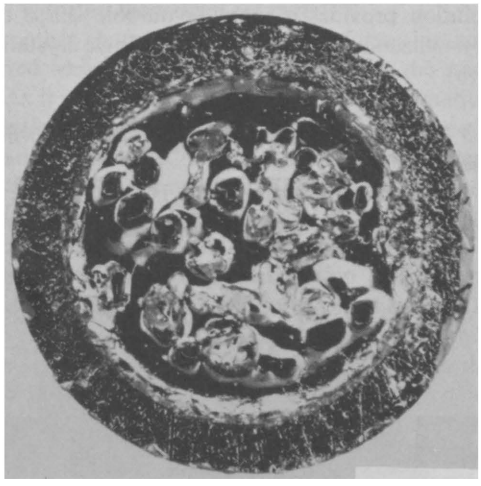


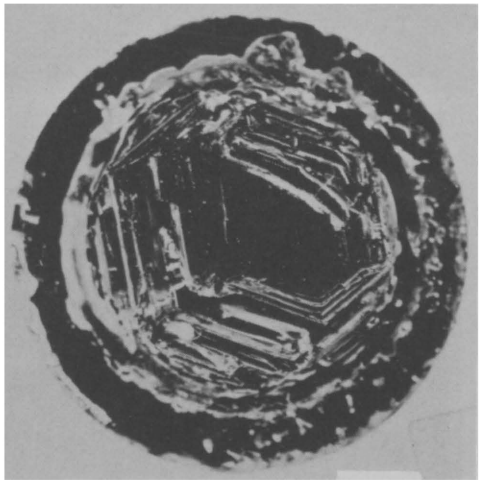
Fig. 6. (a) Temperature dependences of Seebeck coefficients α, thermal conductivity κ, specific resistance ρ and thermo-electric figure of merit Z for space and Earth-grown samples of $Bi_2Te_{2.7}Se_{0.3}$ (HK-1).

Earth sample constancy of the Hall coefficient ratios in longitudinal and cross section was not observed. Besides that the average carrier concentration in these samples was distinguished. It is equal to $\bar{n} = 2.0 \cdot 10^{19}\,cm^{-3}$ and $\bar{n} = 2.7 \cdot 10^{19}\,cm^{-3}$ for space and Earth samples, respectively.

3.2 *The GaP crystals*

3.2.1 *Surface morphology and structural perfection.* The surface morphology and the structural perfection of GaP crystals obtained in microgravity conditions were investigated and described in paper[9]. All space-grown crystals in contrast to the Earth-grown ones, have a well-developed surface relief (Fig. 7a). On the crystal surface HK-4A (Fig. 7b) a dendritic platelet crystal with the large area and well developed facets is seen. This platelet crystal was apparently captured by the surface of the growing layer during a fast furnace cooling. The absence of convective flows in space conditions promotes the formation of dendritic platelets in the solution volume which are larger in comparison with the case of crystal growth on the Earth.





(b)

Fig. 7. (a) Surface of GaP space-grown samples HK-5A; (b) HK-4A crystals.

The crystalline structure of GaP crystals was studied on cuts parallel to the growth axis. The Laue patterns were made on various points of these samples. On Laue patterns obtained from cuts of Earth-grown samples Laue spots, as a rule, were bifurcated, i.e. all layers grown in Earth synchronous experiments have a block structure. At the same time the samples grown aboard the orbital station were single-crystals. On Laue patterns of space-grown samples all Laue spots have a regular shape.

The difference in the degree of defects in these crystals should be mentioned. In all space samples the dislocation density on crystal surface turned out to be of the order of $10^4 \cdot 10^5\,cm^{-2}$ (with the dislocation density in the seed not less than $10^5\,cm^{-2}$). In similar layers grown on Earth the dislocation density was more than $10^6\,cm^{-2}$.

3.2.2 *The analysis of heat and mass transfer.* The distinction in the morphology and structure of crystals can be explained by a different character of the heat and mass transfer process during crystal growth in the space and on the Earth. The detail analysis of the effect of the natural convection on the heat and mass transfer process in Ga–GaP solution based on experimental data was made by means of the boundary layer approximation[4] and mathematical simulation by the simultaneous computer solving of the system of Navier–Stokes, continuity, diffusion and heat conduction equations[10]. The temperature dependence of relative growth rate Vk/G of space and Earth crystals is shown in Fig. 8. The exponential temperature dependence of Vk/G for the crystal growth in the microgravity ($g \approx 10^{-4} g_0$) depicted in Fig. 8 indicates the diffusion mechanism of mass transfer. In case of crystal growth on the Earth by the TSM[4] and SSD method[11, 12] the considerable spread of experimental dots was observed. These dots are above the diffusion curves that shows the presence of the natural convection in the solution leading to

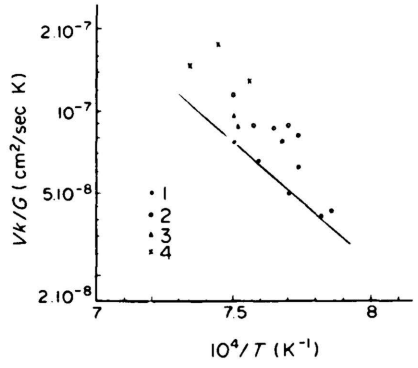


Fig. 8. Temperature dependence of the relative growth rates of GaP crystals (1) space experiments; (2) Earth experiments by TSM; (3) from paper[11]; (4) from[12].

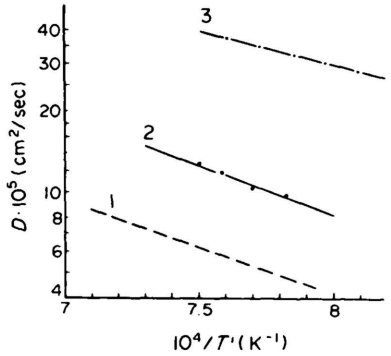


Fig. 9. Temperature dependence of phosphorus diffusion coefficient in liquid gallium (1) from[14]; (2) our data; (3) from[13].

the increase of the crystal growth rate. On the basis of data on the crystal growth rate in microgravity and also on the basis of the results of the investigation of the "Crystal" furnace profile for the growing temperature the phosphorus diffusion coefficients in liquid gallium within the temperature range of 1000–1100°C were determined: $D = 6.83 \cdot 10^{-2} \exp(-0.72\,\text{eV}/T)$[4]. As can be seen from Fig. 9 our curve is below the curve obtained in paper[13] whose authors determined the diffusion coefficient from the data on the phosphorus dissolution rate in liquid gallium with the intensive convection. On the contrary our, diffusion coefficient determined from the crystal growth rate by SSD method[14] are lower though the activation energy almost coincides with our value. Apparently that is a result of averaging of experimental data over a large range of crystal growth temperature.

Knowing exact experimental conditions and the accurate phosphorus diffusion coefficient in the solution the effect of the natural convection on the crystal growth rate can be quantitatively estimated. The effect of the convective flow intensity expressed by the Rayleight criterion Ra on the growth rate is shown in Fig. 10. The degree of the growth rate variation is expressed by the ratio Vk/Vko, where Vk is a real growth rate and Vko is the growth rate in the same thermal conditions without the convection. As seen, the linear dependence of Vk/Vko on $\sqrt{\text{Ra}^{1/4}}$ is observed that conforms to the boundary layer theory[15]. However, with a large length of the zone ($l \approx 5 \cdot 6$ mm) the experimental dots deviate from this curve (Fig. 10). This is apparently associated with the transition from one-vortex to two-vortex structure of the convective flow in the solution existing with the zone thickness of the order of 5–6 mm[10]. So, for a multi-vortex structure of the convection the mass transfer in the solution cannot be described on the basis of a simple boundary layer model.

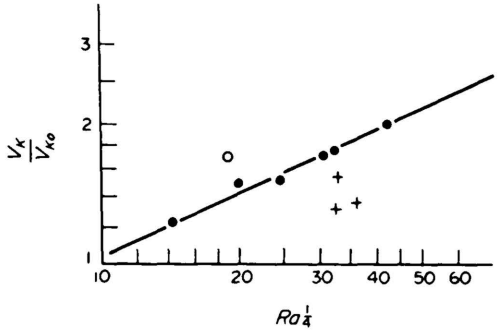
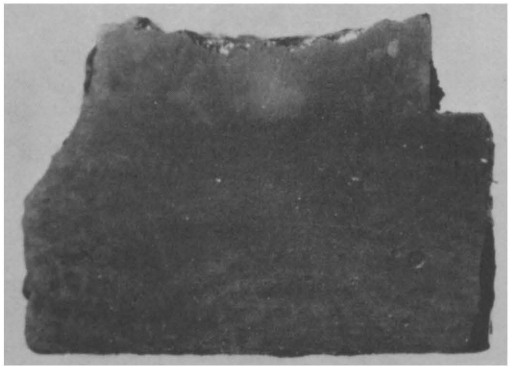


Fig. 10. Dependence of the GaP crystal growth rate on the convection intensity: ○—theoretical calculation; ●—from experiments with zone length $h < 4$ mm; +—from experiments with zone length $h = 5 \cdot 6$ mm (both with unstabilizing density gradient on Earth).

The analysis made in[10] shown the presence of the radial temperature gradient in solution leading to the curvature of the crystallization front and to the appearance of the convection even in case of the density stabilizing gradient for the crystal growth on the Earth. It is probably a main reason promoting the formation and development of blocks and their misorientation (Fig. 11a). In case of crystal growth in space conditions the absence of the convection in the solution provides a more favourable shape of the crystallization front for obtaining single crystals (Fig. 11b).

3.3 *The bubble formation for the crystal growth from the liquid phase*

The bubble formation in Bi_2Te_3–Bi_2Se_3 melt under gravity-free conditions was analysed in paper[16].



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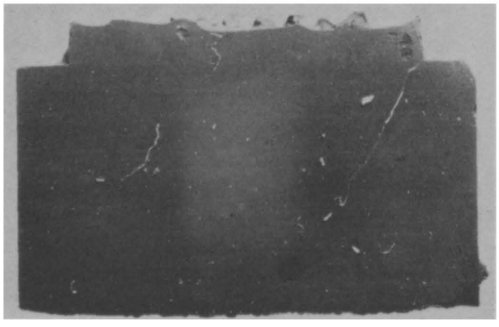


(b)

Fig. 11. (a) Longitudinal section of GaP Earth and (b) space-grown samples.

From the experimental growth conditions and thermodynamic data of Bi_2Te_3–Bi_2Se_3 system the estimate of bubble sizes formed homogeneously in the melt volume was made and the dynamic of their development was considered. For usual growth rate (in our case $Vk \leqslant 5 \cdot 10^{-4}$ cm/sec) the bubbles behave as an impurity with distribution coefficient much less than unit. They are not captured by the crystallization front but pushed aside to the ingot surface and are exhausted forming blow holes on it (Fig. 1a). Apparently, this is a main reason of that the bubbles were observed only on the crystal surface without them inside the ingot, excluding the part of the passive cooling. As it can be seen from Fig. 1(a) larger bubbles were observed in that part of the ingot which melted down before during the heating of the furnace that conforms to the calculation made by us[17]. During the passive cooling there are almost no bubbles on the ingot surface as they were fromen in melt which was fast hardened (Fig. 12). The sizes of such bubbles are of the order of 100–200 μm that agrees with the theoretical estimate[17].

The other character of the bubble formation was observed while growing GaP crystals from the solution. As it was shown in paper[18] bubble formation in solution growth is most likely by heterogeneous nucleation on the seed crystal surface for which very small driving force is required. The calculation shows that for the gallium contact angle on the (111) GaP surface of the order of 110° the free energy of a bubble critical embryo formation can decrease by more than 8 orders of magnitude[19]. Figure 13(a) shows a longitudinal section of the space sample grown at 1060°C. It is seen that all bubbles with the size of 100–300 m are localized on the seed-growing layer interface and have various shapes: round, triangular extended by the growth direction. Such bubbles with less sizes were observed in the earth layers grown with the position of the seed above the solution[9]. These bubbles were apparently formed during heating and gradual wetting of the solution with the seed because of the interaction of the gallium oxide film

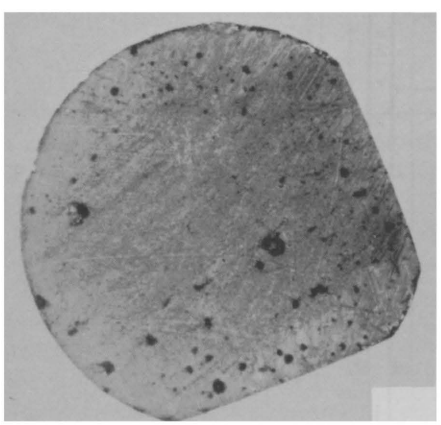
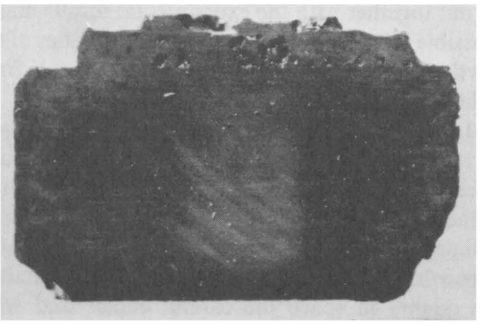


Fig. 12. Cross section containing the bubbles at the end of the HK-1 space-grown ingots.



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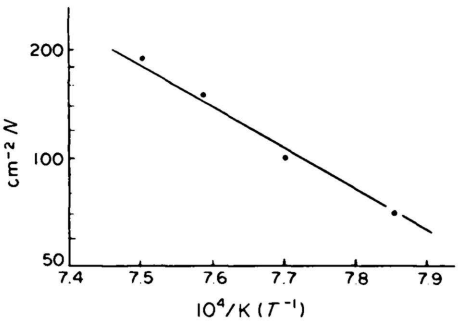

(b)

Fig. 13. (a) Longitudinal section of GaP space sample grown at $T = 1060$°C and (b) the temperature dependence of intensity of bubbles formation on interface Ga–GaP seed.

Ga_2O_3 with gallium releasing volatile monoxide Ga_2O[19]. Therefore, the formation of bubbles decreasing the quality of crystals can be excluded or decreased by preliminary wetting of the seed by gallium and by removing the oxide layer on the solution surface. It should be also mentioned that the bubble formation depends strongly on the growing temperature (Fig. 13b). Its intensity can be decreased by decreasing the growth temperature. The cut of the space sample grown at the temperature $T = 1000$°C is shown in Fig. 11(b) which is almost free of bubbles.

4. CONCLUSIONS

According to the investigation results of the crystals obtained in the HALONG programme the following conclusions can be made.

(1) The crystallization process of $Bi_2(Te,Se)_3$ and GaP crystals from liquid phase differs considerably under Earth and space growth conditions. For the growth of these crystals in space the diffusion mass transfer prevails in the liquid phase. While investigating the crystallization of these systems in space it is possible to determine the diffusion coefficients of components in the liquid phase and also to isolate the effect of the gravity, in particular, the effect of the natural convection on crystal growth process.

(2) The analysis of the mass transfer process, the impurity segregation and the bubble formation car-

ried out together with the experimental results makes it possible to understand better the peculiarities of the growth process of $Bi_2(Te,Se)_3$ and GaP crystals from the liquid phase in space and Earth conditions.

(3) Comparative investigations of crystals grown in space and Earth conditions show that their structural perfection and composition homogeneity strongly depend on the character of mass transfer process in the liquid phase and on the technological parameters of growth process. Therefore for obtaining crystals of high quality in space the careful choice and strict control of growth regime are required.

Acknowledgements—The authors of this paper would like to express their gratitude to the crew of Space Laboratory "Salyut-6" the Vietnamese cosmonaut Pham Tuan and the Soviet cosmonauts L. I. Popov, V. V. Gorbatko and V. V. Ryumin for carrying out the HALONG experiments. They are grateful to Drs. V. T. Khryapov and V. I. Barmin for performing the ground-base experiments.

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