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宇宙での結晶成長実験から

From Crystal Growth Experiments in Space

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1. Introduction

The gravity on the earth has strong effects on the crystal growth. Under the gravity, melt in a crucible is strongly pushed to the wall that results in a stress in the crystal when the melt is solidified. The stress causes the generation of the defects such as dislocations.

The gravity will induce buoyancy driven convection in the melt and the solution from which the crystal is grown. The convective flow gives impurity striations which are generated by temperature fluctuation at the growth front. In space one can eliminate both of such gravity originated effects. In this report, firstly we describe the study of macrostep formation mechanism which was conducted in convection free solution growth. Secondly, contact free Bridgman growth of GaSb is described and the growth of GaSb crystal with low dislocation density is discussed.

2. Crystal Growth Experiments in Space

2.1 Macrostep formation and annihilation under temperature gradient

In a solution growth, it is very often to observe macrosteps on the as grown surface. The macrosteps are generated by the bunch of the atomistic steps during the growth. The reason why the atomistic steps bunch has been unknown. The first explanation (1) was given as that the formation of the macrostep decreases whole surface free energy. The second explanation (2) bases on the assumption that morphological instability happens at the growth interface due to the non-uniform solute diffusion associated with surface undulation. The latter phenomenon has been known classically as constitutional supercooling.

The space experiment conducted by German group^{1),2)} successfully made clear whether (1) or (2) is correct. **Figure 1** shows a cross section of InP crystal grown in space. In space, the grown crystal was rotated to introduce impurity striation so that the local growth velocity was measured. It is important to conduct the growth in space to avoid buoyancy driven convection. Otherwise, rotational striation cannot be observed. It is clearly seen that under temperature gradient, macrostep was generated when the growth velocity was high and disappeared when the growth rate was low. This is a typical behavior of morphological instability in other word the behavior of constitutional supercooling³⁾.

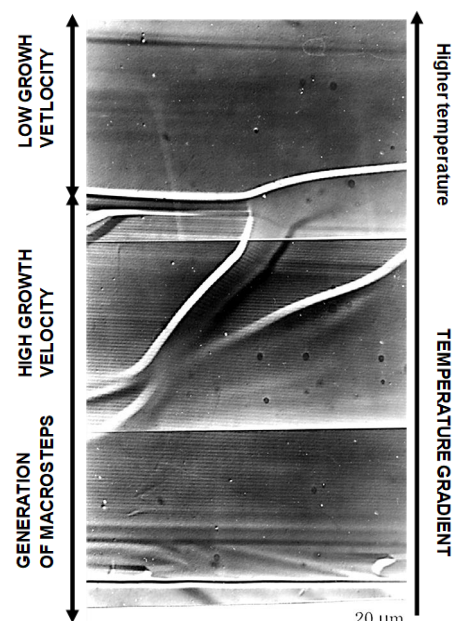


Fig. 1 Cross sectional micrograph of THM grown InP by D-1 mission (1985), K. W. Benz *et al.*

2.2 Bridgeman growth of GaSb by Chinese recoverable satellite

We, the University of Tokyo, had an opportunity to conduct a joint work with Institute of Physics, Chinese Academy of Sciences to grow GaSb single crystal employing Chinese recoverable satellite⁴. We sent four quartz ampoules each of which contained Czochralski grown single crystal of GaSb. Among four samples, only one was successfully re-grown which is shown in Fig.2. The left end shows the un-melted part of the sample which served as seed. Following the seed, growth was conducted with non-contact condition. At the middle of the molten part the melt had contact with the quartz ampoule due to the volume expansion of the melt at the solidification.

Fig. 3 shows X-ray topography image⁵ of GaSb crystal of Fig. 2. In the seed part one sees the impurity striations which were introduced during earth growth. Part 1 shows the place where the growth was conducted at non-contact condition. It was not observed impurity striation, which shows there was no strong buoyancy driven convection. There were only a few dislocations observed.

On the other hand, in the part of contact growth (Part 2 and 3) many kinds of defects were observed being induced such as twins, stacking faults and a high density of dislocations. This has been confirmed by chemical etching experiments which was carried out with the space grown GaSb wafers cut perpendicular to the growth direction⁴. The distribution in the space grown crystal shows typical diffusion controlled one which also shows that there is no strong buoyancy driven convection⁶.

3. Summary

As the examples of the crystal growth in space, two topics (1) study of macrostep formation mechanism utilizing buoyancy driven convection free condition in space and (2) non-contact melt growth of GaSb under microgravity in space were described. Microgravity gives great advantages in studying crystal growth and it is required to expand this opportunity especially with international collaboration.

References

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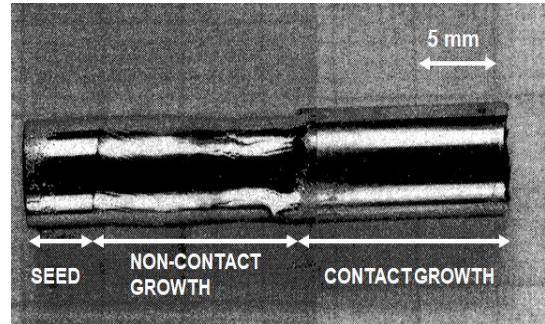


Fig.2 As grown surface of space grown GaSb doped with Te. The surface of non-contact part shows a free surface while the contact part shows mirror like surface of inside crucible wall .

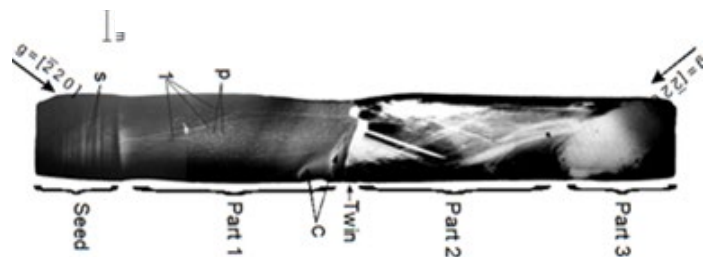


Fig.3 X-ray topography image of space grown GaSb single crystal⁵.

