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### Detached Solidification of Germanium-Silicon Crystals on the ISS

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A series of  $\text{Ge}_{1-x}\text{Si}_x$  crystal growth experiments are planned to be conducted in the Low Gradient Furnace (LGF) onboard the International Space Station. The primary objective of the research is to determine the influence of containment on the processing-induced defects and impurity incorporation in germanium-silicon alloy crystals. A comparison will be made between crystals grown by the normal and “detached” Bridgman methods and the ground-based float zone technique. A schematic of the normal and detached Bridgman processes is shown in figure 1. In both the normal and detached Bridgman processes, the melt is in contact with the ampoule wall. The ampoule is translated with respect to a thermal gradient and the melt directionally solidifies. However, in the detached Bridgman process, a meniscus forms at the bottom of the melt between the crystal and ampoule wall. The size of the gap below this meniscus is typically of the order of 10-100 microns. There are a couple of key angles which govern the process. The contact angle  $\theta$  indicates the degree to which the melt wets the ampoule wall and the growth angle  $\alpha$  is a material property of the system. Typical values for  $\alpha$  are  $10^\circ$ - $25^\circ$  for semiconductors and  $0^\circ$  for metals.

Figure 2 shows the results of a germanium-silicon detached growth experiment on Earth. The upper left-hand image shows the transition from detachment to attachment. Detached portions of the crystal have a smooth and shiny appearance and the attached portions mirror the inner surface roughness of the ampoule container. The upper right-hand image shows an area of the crystal that is completely attached. The surface profilometry measurements are shown at the bottom of the figure. The crystal became detached part of the way through growth and

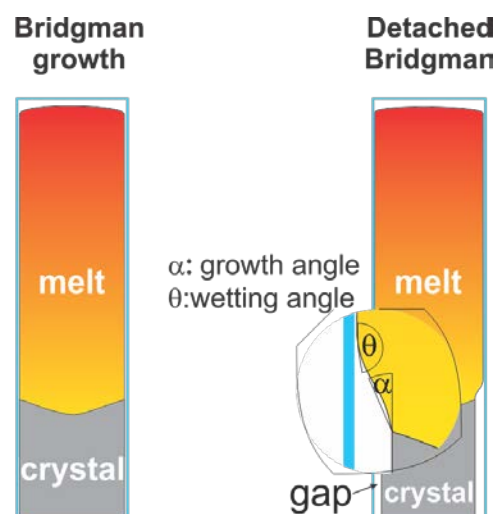
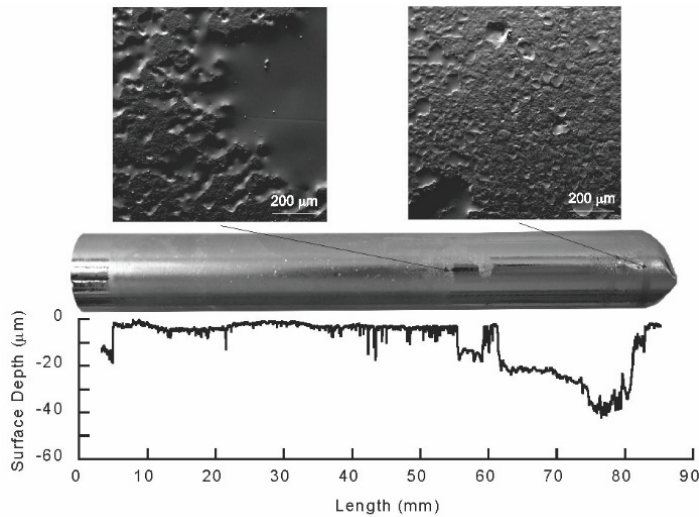


Fig. 1. Schematic drawing showing the essential features of the normal and detached Bridgman crystal growth techniques.



*Fig. 2. The left-hand scanning electron image is an expanded view of the transition between attached and detached regions. The right-hand image is an expanded view of a completely attached region. The y-axis of the plot is the depth of the detachment. The x-axis dimensions of the profilometer measurements also correspond to the dimensions of the macroscopic picture [3].*

the gap width reached about 40 microns. The absence of contact between the crystal and the ampoule during growth leads to significant improvements over crystals grown by the standard methods. Factors leading to such improvements include a reduction in both thermal and mechanical stresses and reduced nucleation of grains and twins at the ampoule wall. Crystals grown detached on the ground exhibited superior structural quality as evidenced by measurements of etch pit density, synchrotron white beam X-ray topography and double axis X-ray diffraction.

Observations of detached solidification date

back to the NASA Skylab mission [1, 2] and were sometimes seen in subsequent microgravity experiments. This is because in microgravity the pressure head in the melt is reduced by six orders of magnitude. Detached growth requires the establishment of a meniscus between the crystal and the ampoule wall and its existence depends on the ratio of the strength of gravity to capillary forces. On Earth, this ratio is large and stable detached growth can only be obtained over a limited set of conditions. Although detached solidification phenomena have been previously observed in microgravity, a systematic microgravity study is required to better understand the process and enable its control on Earth.

In this investigation, a series of 10 GeSi crystal growth experiments will be conducted under various processing conditions. One condition to be varied is the pressure differential across the meniscus. The prevailing theory of detached solidification suggests that detachment is a strong function of this pressure differential and microgravity conditions are required to enable the variation of this pressure over an adequate range. Other parameters to be varied in the flight experiments are the sample material (GeSi or Ga-doped Ge), and the inner ampoule material, which will affect the contact angle.

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- [2] A. F. Witt, H. C. Gatos, M. Lichtensteiger, M. C. Lavine, C. J. Herman, *J. Electrochem. Soc.* 122 (1975) 276-283.
- [3] M. P. Volz, M. Schweizer, N. Kaiser, S. D. Cobb, L. Vujisic, S. Motakef, F. R. Szofran, *J. Cryst. Growth* 237 (2002) 1844-1848.