CdZnTe is one of technologically important II-VI compound semiconductors. CdZnTe has been mainly used as a substrate material for the epitaxial growth of HgCdTe infrared detectors. CdZnTe crystal is typically grown using unseeded, horizontal or vertical Bridgman crystal growth techniques. The Bridgman growth technique has advantages of being easily instrumented and interface curvature can be readily adjusted by changing the temperatures at the top and bottom of the gradient. The disadvantage of the Bridgman growth technique is that the crystal contacts the ampoule wall, which may result in increasing the mechanical stress, impurity level, and defect density in the grown crystal. The disadvantage can, however, be overcome by the detached solidification technique (growth without wall contact).

We have proposed to grow a CdZnTe crystal without wall contact using a unique soft wall technique. The objectives of the proposed research include: (a) understanding the mechanism of detached solidification, designing the furnace, and determining the optimal growth conditions; (b) understanding the role of microgravity in detached solidification; (c) investigating the effect of detached solidification on solidification interface, stoichiometric control and macrosegregation; and (d) studying the effect of detached solidification on stress reduction, dislocation and twinning. An integrated numerical model for detached solidification has been developed combining a global heat transfer sub-model and a wall contact sub-model for the proposed modified Bridgman system. The global heat transfer sub-model accounts for heat and mass transfer in the multiphase system, convection in the melt, macro-segregation, and interface dynamics. The location and dynamics of the solidification interface are accurately tracked by a multizone adaptive grid generation scheme. The wall contact sub-model accounts for the meniscus dynamics at the three-phase boundary, which is similar to the model proposed by Duffar et al. This sub-model has been used to investigate the effects of various geometric parameters, e.g., growth angle, wetting angle, number density, and shape of the finned structure on the CdZnTe detached growth. The global heat transfer sub-model has been used to investigate the effects of the carbon velvet fins on heat and mass transfer in the furnace and detached solidification, and the effects of detached solidification on stoichiometry, micro/macro-segregation under micro- and normal gravity conditions. Simulations have been performed for crystal growth in a conventional ampoule and the designed ampoule to understand the benefits of the detached solidification and its impacts on crystalline structural quality, e.g., stoichiometry, macro-segregation, and stress. The integrated model can be used in designing apparatus and determining the optimal geometry for detached growth in space and on the ground, and the effects of detached solidification on dislocation density and twinning formation will be investigated in the near future.

1. Background and Objectives
Cadmium-Zinc-Telluride (CdZnTe) crystals were grown in unit gravity and in μ-g for comparative analysis in our prior program, Orbital Processing of High Quality Doped and Alloyed CdTe Compound

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Semiconductors. Two Zn alloyed CdTe crystals were grown on USML-1/STS-50 in 1992, and two additional crystals were grown on USML-2/STS-73 in 1995. The Crystal Growth Furnace (CGF) in the seeded Bridgman-Stockbarger crystal growth geometry was utilized on both missions. Crystals grown on USML-1/STS-50 were found to have solidified with partial wall contact due to the near-absence of the hydrostatic pressure in μ-g, the absence of melt volume constraint, a residual g-vector that was not axial, and non-wetting sample/ampoule wetting conditions. Crystals grown on USML-2/STS-73 were grown with a non-axial residual gravity vector, and included: a sample/ampoule identical to the USML-1/STS-73 sample/ampoule, with the addition of a restraining spring to simulate hydrostatic pressure internally; and a tapered ampoule intended to minimize wall contact. A crystal with 2.2 cm without wall contact and 2.8 cm with partial wall contact has been accomplished.

Infrared transmission of all ground and flight samples was found to be 63–66%, very close to the theoretical 66%, suggesting very good stoichiometric control. Longitudinal macrosegregation, calculated using scaling analysis, was predicted to be low. Nearly diffusion controlled growth was achieved even in unit gravity and macrosegregation data could be fit with a diffusion controlled model. Radial segregation was monitored and was found to vary with fraction solidified, particularly through the shoulder region, where the sample cross-section was varying significantly. In regions where a steady-state was established, the radial segregation was invariant, within our experimental measurement error. Ground samples exhibited a fully developed (111)[110] dislocation mosaic structure, whereas dislocations within the flight samples were discrete and no mosaic structure was evident. Dislocation etch pit density results were confirmed using etch pit microscopy, transmission synchrotron white beam and transmission monochromated beam topography. Dislocation density was quantitatively reduced from 75,000 (1-g)±50% to 800 (μ-g)±50%. The low defect density is thought to have resulted from the near absence of hydrostatic pressure, which allowed the molten boule to solidify with little or no wall contact. This minimized the stresses during solidification and transfer of hoop stresses during post-solidification processing.

In regions where solidification had occurred without wall contact the free surfaces evidenced virtually no twinning, though twins reappeared in the flight samples in regions of wall contact for the tapered USML-2 sample. Twinning was pervasive in the ground samples. These results were confirmed using optical microscopy and synchrotron x-ray white beam and monochromatic beam topography. The reason for the dramatic reduction in twins in regions without wall contact is without explanation, though they are thought to be largely surface nucleated.

Our efforts to extend the processing over larger regions of the crystal on USML-2/STS-73 was partially successful, using a tapered ampoule with the wall “falling away” from the triple junction. This increased the material solidified without wall contact. The goal of this research is to further our investigation of the influences of gravitationally-dependent phenomena (hydrostatic and buoyant) on the growth and quality of doped and alloyed CdZnTe. A modified seeded Bridgman-Stockbarger technique with a soft ampoule has been designed. We will study the influences of damping the gravitationally-dependent buoyancy convection on chemical homogeneity, with particular attention to chemical distribution in close proximity to free surfaces. This will address the issue of thermo-capillary flow at free crystal growth surfaces, and the influences of such flows on chemical redistribution and surface generation of defects. More to the point, however, the near-elimination of hydrostatic pressure will enable crystals to be grown in a novel soft ampoule geometry that would be impossible terrestrially. These experiments will allow us to test the origins and theory of twinning and the origins and distribution of dislocations (propagation,
annihilation, multiplication, and redistribution) in this important detector material. The objectives of the proposed research include: (a) understanding the mechanism of detached solidification, designing the furnace, and determining the optimal growth conditions; (b) understanding the role of microgravity in detached solidification; (c) investigating the effect of detached solidification on solidification interface, stoichiometric control and macrosegregation; and (d) studying the effect of detached solidification on stress reduction, dislocation and twinning.

2. Detached Growth Technique
CdZnTe is one of technologically important II-VI compound semiconductors. CdZnTe has mainly used as a substrate material for the epitaxial growth of HgCdTe infrared detectors. CdZnTe crystal is typically grown using unseeded, horizontal or vertical Bridgman crystal growth techniques. The Bridgman growth technique has advantages of being easily instrumented and interface curvature can be readily adjusted by changing the temperatures at the top and bottom of the gradient. The disadvantage of the Bridgman growth technique is that the crystal contacts the ampoule wall, which may result in increasing the mechanical stress, impurity level, and defect density in the grown crystal. The disadvantage can, however, be overcome by the detached solidification technique (growth without wall contact). The first report about detached growth was one of the results of the Skylab missions. In the intervening years, detached Bridgman growth has been observed in many microgravity experiments. Larson et al. provided extensive characterization of partially detached growth of CdZnTe crystals with respect to dislocation density, grain structure, infrared transmittance, and residual stress and strain. They demonstrated that the positive influence of detachment on the crystal quality. When detached solidification occurs, the structural quality of the crystal is improved (fewer dislocations, lower residual strain, and fewer grains), probably owing to the disappearance of stresses exerted by the crucible/ampoule on the crystal during the cooling of the crystal and reduction of heterogeneous nucleation sites.

Although tremendous advancements have been achieved in detached growth techniques, the mechanism is not well understood. Detached solidification is particularly prevalent among semiconductors. Growth angle is virtually zero for a metal and high for a semiconductor; it is therefore possible that growth angle plays an important role in this phenomenon. From the literature surveyed, it can conclude that the appearance of detached solidification seems related to crystal growth angle, wetting angle, crucible roughness, and gas pressure. In summary, detached growth will more likely appear in the following conditions:
- Large wetting angle between the melt and the crucible material,
- Large growth angle between the meniscus and the crystal surface, and
- Small gas pressure difference between the gap and the top of the melt.

All three factors affect the shape and dynamics of the meniscus and three phase boundary. Wilcox, Regel and co-workers and Duffar et al. have proposed different growth mechanisms. The model of Wilcox, Regel and co-workers was based on force balance between surface tension and gas pressure in the gap separating the charge and ampoule. The detailed growth model was developed accounting for segregation of the gas at the solidification interface, evaporation at the melt/gas surface, and bulk transport by the buoyancy-induced convection. The flux of the evaporating gas was related to gas pressure providing the necessary input for the calculation of the gas/melt meniscus shape. The results indicated that reduction of convection in the melt increases the gas content in the melt at the growth front leading to a more stable detached growth condition. Duffar et al. proposed that the surface roughness and growth angle of the ampoule were the primary reasons for detached solidification, and conducted numerical simulations aimed
at identification of the required surface roughness to achieve detached growth. Simulations focused on the
kinematics of force balance at the ridges connecting the melt to the rough crucible.

We propose to use a cylindrical ampoule with an internal ‘multiple fin’ structure that will support the liquid
in microgravity with minimum or no wall contact. A typical carbon fiber ‘velvet’ structure of this type is
shown as Fig. 1. The diameter (strength and rigidity), number density, fiber end shape and wetting angle
relative to CdZnTe, and velvet material can be engineered. It has been made into ampoule configurations.
CdZnTe does not wet Carbon, so it should resist penetrating the velvet. Also shown in Fig. 1 is that a low
density, non-wetting fluid (H$_2$O), that expands on solidification, can be solidified without penetrating the
fibers. This is what we propose to do in microgravity with CdTe. We will use the multifin annular regions
for active atmosphere stoichiometric control, and fluid/crystal support. The advantages of the proposed
design include stoichiometric control and meniscus control, since the vapor can be transported freely
between the fins and contacts the melt fully.

In this proposal, we will develop a numerical model which can be used to understand the difference
between crystal growth in a smooth ampoule and in a soft-wall ampoule and determine the optimal fin
structure for the maximum possibility of detached growth of CdZnTe. A length of crystal will be grown
and then the velvet will be discontinued for a length less than the Rayleigh instability length, at which
point the velvet will be reintroduced. Schematic of such ampoule is shown in Fig. 1. This is case I, where
the twin density will be tested with and without contact, with a constant geometry. Two runs are proposed.
The first will be solidified with a concave interface, which should deter facet formation and twinning.
The second will be run with a convex interface, which should promote facet formation and twinning. Our
second experiment series, Case II, will employ the same ampoule type, except with connected sections
with different inner diameters. The connecting fluid sections will reproduce the shouldering region of a
Czochralski geometry. Fluid statics will determine the meniscus shape, which will be predicted by our
model. An increasing diameter and a decreasing diameter will be investigated with concave and convex
interface shapes, as in Case I. This will offer unique insight into the twinning problem.

3. Integrated Model in Bridgman Growth System
In order to improve the electronic properties of semiconductor crystals produced from a liquid phase, it
is essential to reduce the number of defects caused by convection-induced movement within the liquid,
such as growth oscillation or longitudinal or radial differences in chemical composition. Crystal growth
experiments performed in microgravity have shown that, as forecast by theoretical considerations, homogeneous solutal distribution profiles can be obtained nearly without convection. Detached solidification in microgravity results in a crystal surface state without any apparent relation to the surface state of the crucible/ampoule. When detached solidification occurs, the structural quality of the crystals is improved. Its appearance seems related to numerous factors: crystal growth angle, wetting angle, and crucible roughness.

Figure 2. (a) Schematic of a modified Bridgman growth system. (b) an enlarged view at the three-phase boundary. (c-e) Simulation results for the designed experiment.

Major progress has been made in the simulation of Bridgman and other crystal growth from the melt techniques. The current research focuses on high resolution three-dimensional transient simulation, convective heat and mass transfer, gravitational and thermal stress, interface morphology and dynamics, free surface deformation, interaction between the ampoule and crystal, correlation between the defect and stress, micro- and macro-segregation and so on. Major progress has been made in the simulation of Bridgman growth of CdTe and CdZnTe crystals. To simulate detached growth in a Bridgman system, Popov et al. have assumed that a gap forms between the solid and the ampoule wall and a meniscus forms between the wall and the edge of the freezing interface. According to their paper, the dissolved gas is transported into the gap across the meniscus, affecting the pressure in the gap and the gap width. A numerical model was developed to account for heat transfer in the multiphase system (solid, liquid and gas), convection in the melt, and transport of dissolved gas. The locations of the solidification interface and meniscus are tracked explicitly. The stability and heat transfer were investigated.

A state-of-the-art computer model, MASTRAPP (Multizone Adaptive Scheme for TRAnsport and Phase-change Processes) will be used for the numerical simulation. The model solves governing equations of mass, momentum, energy and chemical species to predict the flow and heat transfer in a system. The numerical scheme is capable of capturing the interface shape and location efficiently and accurately since the interface velocity and shape are directly related to interface instability, and defect generation. The model has been extensively used for modeling and simulation of crystal growth. The MASTRAPP can account for global heat and mass transfer in a crystal growth system. The computer code can also predict the thermo-elastic stress in as-grown crystals based on the temperature field history. It can help in correlating the defect generation in the crystal to growth conditions. Transient solute or impurity transport...
transport in the melt is also incorporated in the model to predict the macro-segregation of dopants and alloying elements. For the proposed system, the solid/melt interface and meniscus were tracked explicitly using the multizone adaptive grid generation scheme. The finned wall were modeled by straight fins of uniform or non-uniform cross section. The heat transfer of the fins was solved analytically for a single fin or modeled for interaction between the fins. The analytical and numerical results were integrated into the global/dynamical model. Figures 2(a-b) show the schematic diagram of the designed Bridgman system, and Figs. 2(c-e) show the shape of solidification interface and temperature distribution in this system. In this simulation, the soft wall is modeled as a solid layer attached to the crucible with different thermal conductivity, which depends on the number density of the finned structure.

Duffar et al.\textsuperscript{1} have proposed a mechanism to explain detached solidification on a rough ampoule in space. A similar mechanism is developed for the current soft wall ampoule. A planar-front solidification of the melt in contact with the roughness tip of the crucible is assumed at \( t = 0 \). The hydrostatic pressure within the melt due to the gravity is assumed to be negligible, so that the melt-gas interface has a large radius of curvature, e.g., the melt-gas interface is flat. Detached growth mechanism is proposed as follows: at time \( t = \Delta t \), the growth began and the interface moved a distance of \( \Delta x = \Delta t \times U \), where \( U \) is the growth rate. The angle between the solidifying direction and the melt-gas interface adhering to the second roughness tip is equal to the growth angle \( \alpha_g \) (Fig. 3a). This planar-front solidification continues in this way until the angle between the melt-gas interface and the second roughness tip reaches the wetting angle (contact angle) \( \theta_w \) (Fig. 3b). At this point, the melt detaches from the roughness and the planar-front solidification repeats the same procedure until the melt attached to the next roughness tip (Fig. 3c).

![Figure 3](image)

**Figure 3.** Detached solidification mechanism for a Bridgman crystal growth using a rough ampoule.

Based on above mechanism, Figure 4(a) shows the numerical results of detached solidification for various growth angles. Steady-state solution can be reached after about 15 fins, and only steady-state portion of the solution has been shown in Fig. 4(a). It can be seen that detached gap width changes dramatically as the growth angle changes. From numerical results, it is clear that detached thickness is dependent on growth angle, wetting angle, and gap width and shape of the fins. It is proved that a large wetting angle and a large growth angle do promote detached growth. We can also conclude that a large gap width, \( D \), may also be benefit for detached growth in space experiment. Other interesting conclusions include, if the growth angle \( \alpha_g \) is too small, for example, less than 10\(^\circ\), the detached growth will not happen. Since the growth angle of CdZnTe cannot be changed, to promote detached growth, the number density of the fins should be low and the wetting angle should be high. The shape of the fins has minor influence on detached growth.
For detached growth on the ground, the effect of gravity has to be considered. The melt-gas interface will not be flat. We will introduce a new parameter, the melt-gas interface curvature, $R = \frac{\sigma}{\rho L g h}$, where $\sigma$ is the surface tension (kg/s$^2$), $\rho_L$ is the density of melt (kg/m$^3$), $g$ is the gravity acceleration (m/s$^2$), and $h$ is the height of melt (m). When the following parameters $\sigma = 0.72$, $\rho_L = 5680$, $g = 9.81$, $h = 0.1$, are chosen, $R = 1.29 \times 10^{-4}$ m is calculated. It means that the gap width of the fins should be much smaller than 129µm in the ground experiment. Otherwise, the melt will penetrate in the fins and attach the crucible. Figure 4(b) shows the effect of $R/D$ on detached growth, in which the roughness gap $D$ increases from 7µm to 26µm.

4. Gravity Effects

A sessile drop is used to study the roles of wetting angle (contact angle) and finned structure on detached growth. Since surface tension of a CdZnTe melt is not available, the surface tension of silicon, 720 dyne/cm, has been selected. The surface tension of most semiconductors is about 10 times larger than that of water. Based on this value, the Bond number, $Bo=0.033$, corresponds to a spacing of 1mm between fins. The shapes of sessile drop at different melt volumes are shown in Fig. 5. In the designed ampoule, the spacing between the fins is much smaller than 1mm. Spherical shape of meniscus can therefore be assumed in the ground experiment since the Bond number is much smaller than 0.033. The assumption used to obtain Fig. 4(b) is therefore reasonable. Equilibrium contact angles of 180 degrees means that the melt is total dewetted from the ampoule. In this case, a drop may cover several fins.

5. Heat Transfer in the Designed System

The geometric configuration of the modified Bridgman system is shown in Fig. 2. The molten CdZnTe is contained in a cylindrical ampoule with soft-finned wall. During a typical seeded Bridgman-Stockbarger growth of CdZnTe, crystal growth is accomplished by establishing isothermal hot zone (1175°C) and cold zone (980°C) temperatures with a uniform thermal gradient (35°C/cm) in between. Having seeded the melt, the sample is thermally equilibrated and the sample is directionally solidified at a constant velocity (1.6 mm/hr) by moving the furnace and thermal gradient down the length of the stationary sample, ampoule, and safety cartridge. During detached growth, a gap is formed between the as-grown crystal and the ampoule wall and a meniscus is formed between the edge of the freezing interface and the fin. The vapor can be transported freely into the gap through the multiple fins. CdZnTe crystal exists
as a semiconductor in the liquid phase, and molten CdZnTe exhibits very low thermal conductivity. This feature, coupled with rather high viscosity, leads to the molten state of the material being characterized by a moderate Prandtl number of $\Pr = 0.406$, a value that is roughly thirty times larger than that of most semiconductors, such as Si, GaAs, and InP. A value of order one implies a strong two-way coupling between thermal and momentum transport, which distinguishes the behavior of Bridgman CdZnTe growth from growth of other semiconductors.

To grow a 5.0 cm diameter CdZnTe crystal, the Grashof number $Gr = 1 \times 10^5$ is equivalent to temperature at the hot zone $T_h = T_f + 21.43$ using the physical properties of CdZnTe. Noted that $T_h = T_f + 83$ is used in the USML-2 experiment. It is safe to say that the Grashof number will vary from $5 \times 10^4$ to $10^6$ for the designed system. A set of baseline parameters, $Pr = 0.406$, $Gr = 1 \times 10^5$, $St/Ste = 1$, and thermal conductivity of the finned structure, $K_f = 8.5$, are selected to investigate the sensitivity of the fin structure on detached solidification. Parametric study was performed to examine the effects of governing parameters, e.g., the Grashof and Prandtl numbers, thermal conductivity of the multiple fin structure, and the position of the solidification interface on detached growth. Numerical simulations have been performed for the upper and lower bounds of the parameter. Results show that convection is very strong for the Grashof number of $10^6$. The fluid and temperature patterns are somewhat similar to those in a tall cavity or cylinder for the $Pr$ number of unity. The flow field is weaker and the interface is less curved if the Grashof number is reduced. It can be concluded from numerical simulations that the interface shape is mainly determined by the melt convection in the designed system. This conclusion is different from the Bridgman growth of other semiconductor materials with low Prandtl number. Convection effects are much more significant in the growth of CdZnTe due to the strong coupling between fluid flow and temperature distribution. The crystal growth will therefore be very different in space and on the ground.

The effects of thermal conductivity of a fin structure on fluid flow, temperature distribution and interface shape are presented in Figs. 6(a), 6(b) and 6(c) for three different values of thermal conductivity of the fin structure, $K_f = 0.907, 8.5,$ and $85$, respectively. The fluid flow is weaker if thermal conductivity of the fin structure is lower. However, the effect of thermal conductivity of the fin structure on the interface shape is not significant. This conclusion is not surprising since conductivity of CdZnTe crystal is very low. The
effect will be much significant if thermal conductivity of the growing crystal is higher. More parameter studies have been performed, which can be found in the published papers.  

Figure 6. Effects of thermal conductivity of the fin structure on fluid flow, temperature field, and interface shape in a modified Bridgman growth system with \( Pr=0.406, Gr=1.0e6, \text{Ste}/\text{Ste}_t = 1, K_s = 9.07 \times 10^{-1}, K_{f}=1.085, K_a=85. \)

6. Zn Concentration Distribution In the Designed Furnace
The effects of the Grashof number on Zn concentration distribution in the CdTe melt are investigated. The change of melting temperature with composition is considered though phase-diagram. Numerical results are presented in Fig. 7. The concentration boundary layer thickness at the center of the melt increases when the Grashof number increases from zero to \( 10^3 \), and decreases sharply when the Grashof number increases from \( 10^3 \) to \( 10^4 \). Space experiments showed that nearly diffusion controlled growth were achieved and the radial segregation was found to vary with fraction solidified, particularly through the shoulder region, where the sample cross-section was varying significantly. In regions where a steady-state was established, the radial segregation was invariant. Numerical results show that the radial segregation will also vary significantly when the solidification interface passes through the shoulder region, where the Grashof number changes rapidly, in the ground experiment.
Figure 7. Concentration distributions in the ampoule for Sc = 41.5, k = 1.35, and (a) Gr=0, (b) Gr=1×10^3, and (c) Gr=1×10^5 in a modified Bridgman growth system.

Figure 8. Effects of the Marangoni number on fluid flow and temperature distribution for Gr=1×10^5.

7. Marangoni Convection during Detached Growth
Duffar et al.\textsuperscript{19} and Levenstam et al.\textsuperscript{17} have pointed out that Marangoni convection will be important in fully or partially detached growth. To model the partially detached free surface, Levenstam et al.\textsuperscript{17} have
derived a tangential velocity boundary condition, which is a function of the fraction of free surface and the temperature gradient over detached region. Assuming that two-dimensional assumption is still valid and the equivalent Marangoni number can be used, the effects of Marangoni convection on the interface shape and temperature distribution are illustrated in Fig. 8. In this calculation, the Marangoni number is determined by the size of the ampoule and temperature difference between the hot and cold zones. As the Marangoni number increases, temperature gradient in the melt near the interface increases. It makes the solidification interface move to the cold side. The shape of solidification interface has also varied. Furthermore, the cell above the interface becomes much stronger as the Marangoni number increases. Consequently, Zn concentration distribution and radial segregation will vary significantly.

8. Conclusions
A detached growth mechanism has been proposed, which is similar to that proposed by Duffar et al.\(^1\) and used to study the current detached growth system. From numerical results, we can conclude that detached growth will more likely appear if the growth and wetting angles are large and meniscus is flat. Detached thickness is dependent on growth angle, wetting angle, and gap width and shape of the fins. The model can also explain why the detached growth will not happen for metals in which the growth angle is almost zero. Since the growth angle of CdZnTe cannot be changed, to promote detached growth, the number density of the fins should be low and the wetting angle should be high. Also, a much smaller gap width of the fins should be used in the ground experiment and the detached gap width is much smaller. The shape of the fins has minor influence on detached growth. An integrated numerical model for detached solidification has been developed combining a global heat transfer sub-model and a wall contact sub-model. The global heat transfer sub-model accounts for heat and mass transfer in the multiphase system, convection in the melt, macro-segregation, and interface dynamics. The location and dynamics of the solidification interface are accurately tracked by a multizone adaptive grid generation scheme. The wall contact sub-model accounts for the meniscus dynamics at the three-phase boundary. Simulations have been performed for crystal growth in a conventional ampoule and a designed ampoule to understand the benefits of detached solidification and its impacts on crystalline structural quality, e.g., stoichiometry, macro-segregation, and stress. From simulation results, both the Grashof and Marangoni numbers will have significant effects on the shape of growth front, Zn concentration distribution, and radial segregation. The integrated model can be used in designing apparatus and determining the optimal geometry for detached solidification in space and on the ground.

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