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PI Name:	Dunand, David Ph.D.		
Project Title:	Microstructure Evolution in Freeze-Cast Materials		
Division Name:	Physical Sciences		
Program/Discipline:			
Program/Discipline Element/Subdiscipline:	MATERIALS SCIENCEMaterials science		
Joint Agency Name:		TechPort:	No
Human Research Program Elements:	None		
Human Research Program Risks:	None		
Space Biology Element:	None		
Space Biology Cross-Element Discipline:	None		
Space Biology Special Category:	None		
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Comments:			
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No. of PhD Candidates:	1	No. of Master' Degrees:	0
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No. of Bachelor's Candidates:	3	Monitoring Center:	NASA MSFC
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Flight Program:	ISS		
Flight Assignment:			
Key Personnel Changes/Previous PI:			
COI Name (Institution):	Voorhees, Peter Ph.D. (Northwestern University)		
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Task Description:

Freeze-casting is a novel technique that utilizes ice as a fugitive space holder to fabricate a diverse variety of materials exhibiting elongated, aligned pores. Experimental studies in microgravity inherently simplify the freeze-casting system by minimizing gravity-induced forces that contribute to its complexity, e.g., sedimentation, buoyancy, and natural convection. Freeze-casting has the potential to produce porous products with specific microstructure including net- and complex-shaped products, provided solidification conditions are properly controlled. Moreover, freeze-casting holds significant promise as an in situ resource utilization technique for space-based materials processing, thus increasing the reliability and safety of access to space while also decreasing overall costs. An improvement in scientific knowledge entails robust and predictive control of materials for a wide variety of applications, thus enabling optimized fabrication on Earth, on planetary surfaces (Moon and Mars), and in orbit.

Rationale for HRP Directed Research:

Research Impact/Earth Benefits:

Freeze-casting has the potential to produce porous products with specific microstructure including net- and complex-shaped products, provided solidification conditions are properly controlled. Moreover, freeze-casting holds significant promise as an in situ resource utilization technique for space-based materials processing, thus increasing the reliability and safety of access to space while also decreasing overall costs. An improvement in scientific knowledge entails robust and predictive control of materials for a wide variety of applications, thus enabling optimized fabrication on Earth, on planetary surfaces (Moon and Mars), and in orbit.

Task Progress:

Solidifying naphthalene-based particle suspensions using a typical freeze-casting set-up (i.e., a mold containing suspension that is temperature controlled at the top and bottom faces providing control over the overall thermal gradient in the sample, but not the thermal gradient within the melt zone, specifically) resulted in disordered structures. When we attempted to solidify the same materials using a Bridgman furnace, we obtained directional microstructures; thus, we concluded that the disordered structures we obtained using the basic freeze-casting set-up likely resulted from an inadequate control over solidification parameters. Solidification experiments are now being conducted exclusively using a Bridgman furnace. The motor driver on the furnace has been updated for these experiments using a DM542T digital stepper driver with 16 degrees of microstep resolution, corresponding to 200-25,600 pulses per revolution for the system. This provides attainable furnace translation velocities ranging from 0.2 to >1,000 μm·s^(-1), which is consistent with the growth velocities offered by the Pore Formation and Mobility Investigation (PFMI) (1-100 µm·s^(-1)). Optical micrographs showing the microstructure of copper freeze-cast materials obtained by directionally solidifying 5 vol.% Cu particles suspended in naphthalene that contained 0.4 wt.% dissolved, AOT surfactant (1 wt.% AOT with respect to Cu) were provided in our annual technical report to NASA; structures were solidified using a furnace translation velocity, $V=80~\mu m\cdot s^{-}(-1)$ and thermal gradient, $G=35^{\circ}C\cdot cm^{-}(-1)$. Plate-like microstructures are observed for all samples. The microstructure predicted by You et al. for the solidification condition is "spears" (i.e., presence of significant dendritic features that extend into neighboring dendrites, producing a cellular-type structure after sublimation of the solidified fluid). [Ed. Note: See Reference.] The images show that, while significant dendritic features are present, a wall-type structure is retained.

Reference: You J, Wang Z, Worster MG. Controls on microstructural features during solidification of colloidal suspensions. Acta Materialia. 2018 Sep 15;157:288-97. https://

Bibliography Type:

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Articles in Peer-reviewed Journals

 $Lloreda-Jurado\ PJ,\ Wilke\ SK,\ Scotti\ K,\ Pa\'ul-Escolano\ A,\ Dunand\ DC,\ Sep\'ulveda\ R.\ "Structure-processing\ relationships of freeze-cast iron foams fabricated with various solidification rates and post-casting heat treatment." Journal of Materials Research. 2020\ Jul 20;35(19):2587-96. <math display="block"> \underline{http://dx.doi.org/10.1557/imr.2020.175}\ ,\ Jul-2020$