Fiscal Year:	FY 2019	**Task Last Updated:**	FY 08/12/2019
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 SCIENCE--Materials 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:			
Project Type:	FLIGHT	**Solicitation / Funding Source:**	2015 NNH15ZTT002N MaterialsLab Open Science Campaigns for Experiments on the International Space Station
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No. of Post Docs:	0	**No. of PhD Degrees:**	0
No. of PhD Candidates:	1	**No. of Master' Degrees:**	0
No. of Master's Candidates:	0	**No. of Bachelor's Degrees:**	1
No. of Bachelor's Candidates:	1	**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:	[Ed. note (August 2019)--provided to Task Book in August 2019; reporting covers 11/06/2017 – 11/06/2018] This research represents the first microgravity study of quasi-steady state solidification behavior in the freeze-casting process. Given the wide range of typical processing parameters and great number of research-worthy questions that remain unanswered about the technique, an exhaustive literature review was conducted to aid in experiment design. Data linking processing conditions to microstructural characteristics and mechanical properties were extracted from ~900 freeze-casting papers and a systematic analysis of these data was conducted. In accordance with the aim of this program, we created a public freeze-casting data repository (http://www.freezecasting.net/) in an effort to facilitate broad dissemination of relevant data to freeze-casting researchers, promote better informed experimental design, and encourage modeling efforts that relate processing conditions to microstructure formation and material properties. A description of the resulting SQL database/website and results of our analysis were published in a review article in Progress in Materials Science [see Bibliography section]. Typical processing parameters that have been identified will be utilized during experiment design to ensure maximum generalizability of these results. Experimental data from the database will also be utilized to test models developed during this project. Succinonitrile (NC(CH2)2CN; SCN) was chosen as the suspending fluid for freeze-casting test suspensions due to: (i) known compatibility with the Pore Formation and Mobility Investigation (PFMI) apparatus [Grugel et al., 2012], (ii) ease of sample transport (the melting point of SCN is ~58°C; thus, transport of test suspensions and solidified samples requires minimal environment control), and (iii) system simplification. It was determined that simplifying the system to the largest possible extent would offer the greatest degree of fundamental knowledge necessary to improve the understanding of microstructural formation and would also offer the opportunity to validate and improve existing freeze-casting models. This fundamental basis shall provide a basis upon which future microgravity work can build. Unlike water, which is the most-often utilized fluid in freeze-casting studies, SCN exhibits a linear temperature-density relationship within the temperature range of interest; thus, a density inversion during solidification is avoided. SCN has not been reported as a suspending fluid for use in freeze-casting suspensions systems. Previous research has shown that anisotropic solidification behavior of suspending fluids is a necessary, but insufficient criterion, for attaining directional pore structures for particle-based suspension systems [Naviroj et al., 2017]. We conducted preliminary tests to verify the feasibility of attaining directional microstructures using SCN-based particle suspensions. Directional microstructures were confirmed via scanning electron microscopy investigation of the fractured surface of a titanium/SCN freeze-cast structure where 20 vol.% titanium particles (20 µm size) was suspended in molten SCN and solidified under the presence of a thermal gradient. There are two main limitations of our preliminary demonstration, including: (i) constant cooling was utilized and the solidified SCN was not sublimated from the sample and sintering steps were not carried out. For the former issue, one side of the molten SCN suspension was cooled using a constant cold plate temperature (~20°C) while the other side was held at a constant warmer temperature (~65°C), whereas controlled cooling will be utilized during experimental operations as it provides greater control over microstructures templated. With regard to the second issue, our previous freeze-casting projects have mainly utilized water as the suspending fluid. In such cases, frozen samples are sublimated using a conventional freeze-dryer. The physical properties and toxicity of SCN necessitate the development of a new sublimation procedure. Progress toward completing these tasks is described below. We developed an apparatus that will enable controlled solidification testing and a sublimation apparatus suitable for sublimating SCN from solidified samples. Testing of these systems will be conducted over the next two months. References R.N. Grugel, L.N. Brush, A.V. Anilkumar, Disruption of an aligned dendritic network by bubbles during re-melting in a microgravity environment, Microgravity Sci. Technol. 24(2) (2012) 93-101. M. Naviroj, P. Voorhees, K. Faber, Suspension-and solution-based freeze casting for porous ceramics, Journal of Materials Research (2017) 1-11.
Bibliography Type:	Description: (Last Updated: 11/17/2022)
Articles in Peer-reviewed Journals	Scotti KL, Dunand DC. "Freeze casting – A review of processing, microstructure and properties via the open data repository, FreezeCasting.net." Progress in Materials Science. 2018 May;94:243-305. https://doi.org/10.1016/j.pmatsci.2018.01.001 , May-2018