Fiscal Year:	FY 2024	**Task Last Updated:**	FY 01/03/2024
PI Name:	Pathak, Siddhartha Ph.D.		
Project Title:	Structure, Properties, and Performance of Solder Joints in Terrestrial vs. Reduced-Gravity Environments		
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:	NOTE: PI moved in fall 2020 to Iowa State University from University of Nevada, Reno.		
Project Type:	Physical Sciences Informatics (PSI)	**Solicitation / Funding Source:**	2021 Physical Sciences NNH21ZDA014N-PSI: Use of the NASA Physical Sciences Informatics System – Appendix G
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No. of PhD Candidates:	1	**No. of Master' Degrees:**	0
No. of Master's Candidates:	0	**No. of Bachelor's Degrees:**	0
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COI Name (Institution):	Napolitano, Ralph Ph.D. (Iowa State University, Ames)		
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	The investigation proposed here combines experiments and modeling to elucidate the fundamental mechanisms, phenomenology, and process conditions that govern the integrity and performance of solder joints produced in terrestrial vs. reduced gravity environments, such as the microgravity conditions on board the International Space Station (ISS). The technical research program plans to utilize solder samples from the In-Space Soldering Investigation (ISSI) experiments from the Physical Sciences Informatics (PSI) repository, as well as expand into other non-ISSI solder compositions, and combine space- and ground-based experiments with advanced 3D materials characterization, micromechanical testing, and mesoscale modeling. In particular, the project addresses the formation and persistence of porosity through the reflow/filling/freezing processes and the deleterious effects on microstructure and mechanical properties of the solder joint. It has been established that porosity arising from flux volatilization, which is dispersed		

Task Description:	and expelled from the solder joint under terrestrial gravity, may become entrapped within the freezing solder material under microgravity conditions, given the absence of buoyancy-driven convection. Our overall goals are (i) to advance the current qualitative understanding of this phenomenon into the realm of alloy/process-specific quantitative description and prediction, and (ii) to examine the effects of mechanically and acoustically stimulated flow patterns while assessing their potential effectiveness as porosity mitigation strategies for solder-based fabrication processes in space. Considering a range of potential applications and materials, 3 solder alloys will be investigated, including the ISSI lead-based (Pb-Sn) solders, as well as lead-free (Sn-Ag-Cu and Sn-Au) solders, which have recently shown promise for high-performance joint applications due to their thermal/electrical conductivities and excellent corrosion/fatigue resistance.
Rationale for HRP Directed Research:	
Research Impact/Earth Benefits:	The ongoing project is unique in that it combines both experimental and computational research on solder samples from the microgravity and ground-based ISSI experiments, along with a comparative study on fresh solder samples. The insights gained from this work can be extended to study the effects of fluid interfaces with surface tension variations that are also common for many spaceflight-enabling technologies (e.g., boiling, heat transfer, combustion, welding, brazing, or soldering). We envision that this project will contribute to developing an advanced understanding of multiple processes that typically rely on gravity-driven separations in the terrestrial environment, such as the removal of gas bubbles from a liquid, which must be accomplished by other means in microgravity environments. Furthermore, the microgravity experiments represent a lowest gravity boundary condition. As such, these results could also be useful in predicting solidification behavior on other lower gravity environments (e.g., Moon or Mars). This project has the following research impact: (i) Our project objectives are a timely alignment with current NASA initiatives related to metals joining in reduced gravity environments. The three solder alloys for this work were chosen after extensive discussions with our NASA collaborators, and these alloy systems are currently being considered at NASA as possible candidates for use in the NASA Mars Rover sample return missions. (ii) We also anticipate that the results from this project will inform experiment design responsive to the (future) NASA proposal calls such as the NASA Artemis program, which seeks to land the first woman and next man on the Moon. (iii) The insights gained from this work can be extended to study effects of fluid interfaces with surface tension variations that are also common for many spaceflight-enabling technologies (e.g., boiling, heat transfer, combustion, welding, brazing, or soldering). We envision that this project will contribute towards advanced understanding of multiple processes that typically rely on gravity-driven separations in the terrestrial environment, such as the removal of gas bubbles from a liquid, which must be accomplished by other means in microgravity environments. (iv) the developed gravity-level-specific protocols (GLSPs) for several configurations can be used by future scientists and flight personnel for studying metal joining techniques such as welding, brazing, or soldering under reduced gravity.
Task Progress:	The purpose of this research is to elucidate the fundamental mechanisms, phenomenology, and process conditions that govern the integrity and performance of solder joints produced in terrestrial vs. reduced gravity environments, such as the microgravity conditions on board the International Space Station (ISS). Solder joint porosity is a common but undesirable feature naturally arising from the use of fluxes and is more insidious in soldering joints formed under low-gravity conditions. Commercial soldering processes require the use of fluxes to suppress oxidation and promote wetting, which is necessary for effective solder spreading and joint-gap filling during solder reflow. Under typical soldering conditions, rapid reflow temperatures results in flux volatilization and bubble formation. Under the presence of gravity, liquid solders tend to mix vigorously during the reflow period due to buoyancy-driven natural convection. This is beneficial, as any voids or bubbles that form due to evaporated fluxes are quickly swept to free surfaces and are thus removed from the solder joint. In the absence of gravity, fluid motion is greatly reduced, driven mainly by solidification volume change, thermally induced density gradients, and Marangoni effects. With slower mixing, voids and bubbles are not swept away and become entrapped in the interior of the solder joint upon solidification. This entrapped porosity dramatically degrades the thermal and electrical properties of the solder and severely reduces the mechanical integrity of the joint. Such porosity is seen in conventional (Earth gravity) solder joints but is much more prevalent in solder joints produced under reduced-gravity conditions. As a result, any solder joints made under reduced gravity (such as in-orbit repair of electronic devices, tubing, and mechanical joints) are at risk of having their electrical or mechanical performance characteristics substantially degraded by entrapped porosity, increasing the manifold associated risks of subsequent device failures on astronauts and equipment in the unforgiving environment of space. The ongoing project is being conducted under six major tasks. Each task is composed of integrated experimental -- at Iowa State University (ISU), and Advanced Photon Source (APS) -- and modeling at Iowa State University (ISU). In this work, using four microgravity solders from the In-Space Soldering Investigation (ISSI) study, two ISSI terrestrial solders along with a third set of freshly made terrestrial 40wt%Pb-60wt%Sn solder and 4th set of 50wt%Pb-50wt%Sn solders, significant progress has been made in Task A to Task E. Under these tasks, we report results from the advanced microstructural characterization and resultant micro-to-nano mechanical response of solders in terrestrial vs. microgravity environments, 1g vs. ~1×10-5g under different temperature conditions. In Task A (Year 1), we conducted 3-D Microstructural Characterization of Solder Samples at the Advanced Photon Source. We performed non-destructive tomography to characterize the 3-D distribution of Pb-rich dendrites and voids in the Pb-Sn solder. However, high-resolution tomography scans were limited to sample thicknesses of 700 µm or less so we had to cut the solder sample. We cut them down to 700 µm x 1 mm rectangular sections in order to perform the tomography experiment. In Task B, C and D (Partially completed in Year 1), we polished the solder samples section by section for a detailed microstructural characterization and nanomechanical testing of microgravity and terrestrial solder samples. The detailed analysis of voids distribution in the microgravity and terrestrial samples demonstrated that the number of voids in the ISSI microgravity wire wrap solder is ~13 times higher than the ISSI terrestrial solder and ~5 times higher than the recently prepared terrestrial solder. Additionally, ISSI microgravity wire feed solder demonstrated ~3.5 times higher number of voids (larger in size) than the ISSI microgravity wire wrap solder. The higher number of voids in the wire feed solder demonstrated that the excess amount of flux was remained internal to the liquid as compared to the wire wrap solder. The ISSI microgravity wire wrap solder demonstrated a homogeneous distribution of Pb-rich phase. In contrast to the ISSI microgravity wire wrap solder, ISSI microgravity wire feed solder demonstrated the accumulation of voids and Pb-rich dendrites in one region. This difference in the solder microstructure is probably caused by the strong Marangoni flow in the wire feed solder. Further characterization of this sample revealed that this Marangoni effect was probably caused by the presence of a large void in the wire feed solder, which moved Pb-rich dendrites and voids from

low temperature region to the high temperature region.

Task D: A further consideration, not mentioned in the earlier ISSI report is the substantial effect of aging on the solder microstructure and properties over the past 17 years. Previous investigations have shown that Pb-Sn soldering alloys (due to their low melting temperatures) exhibit grain coarsening and reduced mechanical properties with age, even under ambient conditions (room temperature). Microstructural changes stem from the diffusion of Sn in to the Pb rich regions, creating a finer dispersion of grains as well as voids near phase boundaries. After aging 30 days in ambient conditions, the microstructure and mechanical properties appeared to stabilize. In earlier NASA Physical Sciences Informatics (PSI) reports, we have discussed the aging properties of 40Pb-60Sn solder. In the report, we demonstrate how the microstructure and properties of solder joints change as a function of aging in 50%Pb-50%Sn. We noticed that at room temperature, nanomechanical testing showed a decrease in hardness of the top sample from 0.183GPa on the first day to 0.147GPa by day 90.

Additionally, solder joints in the space environment are subjected to large temperature variations, such as -120 ºC to +25 ºC for the Mars missions, and -157 ºC to +121 ºC outside the International Space Station (ISS). However, as deep space exploration missions become longer and more distant, the solder and other metal joints' current reliability is inadequate for the harsh conditions they encounter. Therefore, we performed in-situ scanning electron microscope (SEM) micro-pillar compression experiments at various cryogenic temperatures to understand the effect of ductile to brittle transition and beta Sn to alpha Sn transformation on the solder properties. Our experimental analysis showed that, as the temperature went down, solder demonstrated higher hardening and strength under the compressive loading conditions. The in-depth data analysis of these experiments is still under investigation to understand the effect of phase transformation on the solder properties.

This project has resulted in two undergraduate projects: 1) Investigate the effect of aging on microstructure and properties of 50Pb-50Sn solders under ambient conditions; 2) Non-destructive 3-D microstructural characterization of Pb-Sn and Sn-Ag-Cu solders. This project has provided two junior undergraduate students (UG) and two sophomore UG students with an opportunity to work on in-space soldering and to get hands-on experience on the lab instruments. In addition, this project has resulted in six presentations at various conferences (e.g., ISSRDC, ASGSR, TMS, NCUR, etc.) and six awards (e.g., Wayne G. Basler Scholarship, ASGSR travel award, FHMP Scholar Award, TMS Acta Materialia scholarship). [Ed. Note: See Bibliography.]

Bibliography Type:	Description: (Last Updated: 02/12/2024)
Abstracts for Journals and Proceedings	Kumar M. "Structure and properties of the terrestrial vs.microgravity solders under extreme conditions of elevated and cryo temperatures. Poster presentation." ISSRDC 2023 (2023 International Space Station Research and Development Conference), Seattle, Washington, July 31 - August 3, 2023. Abstracts. ISSRDC 2023 (2023 International Space Station Research and Development Conference), Seattle, Washington, July 31 - August 3, 2023. , Jul-2023
Abstracts for Journals and Proceedings	Kumar M. "Structure and properties of the terrestrial vs. microgravity solders under extreme conditions of elevated and cryo temperatures." 39th Annual Meeting of the American Society for Gravitational and Space Research, Washington, DC, November 13-18, 2023. Abstracts. 39th Annual Meeting of the American Society for Gravitational and Space Research, Washington, DC, November 13-18, 2023. , Nov-2023
Abstracts for Journals and Proceedings	Kumar M. "Structure and properties of solder joints produced in terrestrial and microgravity conditions." TMS 2023 (The Minerals, Metals, and Material Society 2023 Conference), San Diego, California, March 19-23, 2023. Abstracts. TMS 2023 (The Minerals, Metals, and Material Society 2023 Conference), San Diego, California, March 19-23, 2023. , Mar-2023
Abstracts for Journals and Proceedings	Hellyer S, Ogden CM. "Studying the effects of aging on the structure and properties of off-eutectic 50wt%Pb-50wt%Sn solder joints for in-space applications." NCUR-23 (2023 National Conference on Undergraduate Research), Eau Claire, Wisconsin, April 12-15, 2023. Abstracts. NCUR-23 (2023 National Conference on Undergraduate Research), Eau Claire, Wisconsin, April 12-15, 2023. , Apr-2023