Proceedings of the SPIE Smart Structures and Materials/NDE Symposium, Portland, Oregon, March, 2017

Synchronous Separation, Seaming, Sealing and Sterilization (S4) using Brazing for Sample Containerization and Planetary Protection

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Abstract

The potential return of samples back to Earth from other planetary bodies would be based on planetary protection requirements that vary depending on the type of body [1]. Potential Mars Sample Return would require the protection of our planet from backward contamination. To fulfill this requirement, it would be necessary to implement "break the chain of contact (BTC)" process, where any material reaching Earth would have to be inside a container that is sealed with an extremely high level of confidence. In order to accomplish this, it would be necessary to contain the acquired samples and destroy any potential biological materials that may have contaminated the external surface of the container, while protecting the samples for further analysis. Using brazing, a novel synchronous separation, seaming, sealing and sterilization (S4) process for sample containerization and planetary protection has been conceived and demonstrated. A prototype double-wall container with inner and outer shells and Earth clean interstitial space was used for this demonstration. For potential Mars sample return, the double wall container would be consist of two halves and prepared on Earth. The on-orbit execution would consist of inserting the sample into one of the halves and then mating to the other half and melt the braze material to perform the S4 process. The use of brazing material that melts at temperatures higher than 500^{O}C would assure sterilization of the exposed areas due to pyrolysis since carbon bonds are broken at this temperature. The process consists of two-steps, Step-1: the double wall container halves are fabricated and brazed on Earth; and Step-2: Assembly and brazing the samples on orbit. To prevent potential jamming during the process of mating the two halves of the double-wall container and the extraction of the brazed inner container, a double cone-within-cone approach has been conceived. The results of this study are described and discussed in this manuscript.

Keywords: Sample return, planetary exploration, brazing, synchronous Separation, Seaming, Sealing and Sterilization (S4), break the chain of contact (BTC), containerization, and planetary protection.

1.0 Introduction

Exploring space and determining if we are alone in the Universe has been the goal of humans for generations. The initial missions focused on observing the various bodies in the solar system from space using orbiting or flyby spacecraft. As our capability has increased and reaching other planetary bodies has become feasible, major reports such as the Planetary Science Decadal Survey have prioritized the return of samples of these bodies back to Earth for study [2]. After planetary landing and in-situ sample acquisition and analysis became feasible, Mars and other bodies have become the target of landed missions; the latest Mars rover, Curiosity, landed in August 2012. Tests of returned samples would provide critical information about the composition of the Martian surface, and help us learn as much as possible about the evolution of the martian surface and atmosphere. Returning samples to Earth in a potential NASA mission would require protection of our planet from backward contamination that could be returned with the samples. One of the requirements of such a mission is that if there is a probability of failure higher than 10^{-6}, the mission would be aborted and the sample container would not be returned to Earth [1]. An effective and extremely reliable mechanism would be needed to assure the required high level of planetary protection of Earth in support of potential sample return from Mars and other planetary bodies with potential for habitable environments. The mechanism would have to hermetically seal the returned sample container and perform planetary protection by sterilizing the seal/seam section, and assure that contaminated surfaces are prevented from exposing the outside of the returning container or its transporting vehicle. In order to ensure the success of the mission at the required probability, it would be necessary to "break the chain of contact (BTC)", where any Martian material reaching Earth would have to be inside a sealed container with extremely high level of confidence. Therefore, it would be necessary to contain the acquired samples and destroy any potential biological materials that may contaminate the external surface of the container while protecting the sample for further analysis.

A novel synchronous separation, seaming, sealing and sterilization (S4) process has been conceived and demonstrated that provides sample containerization and planetary protection [3 - 6]. Using brazing [7], prototype double wall containers with inner and outer shells and an equivalent of "Earth clean" interstitial space were designed, produced, and demonstrated. In a potential future mission, the double wall container would consist of two halves and prepared on Earth, while the on-orbit execution would consist of inserting the sample into the inner halves and then mating to the other half and melt the braze material to perform the S4 process. The use of brazing material that melts at temperatures higher than 500ºC would assure sterilization of the exposed surfaces since carbon bonds are broken via pyrolysis at this temperature. The process consists of two-steps, Step-1: the double wall container halves are fabricated and brazed on Earth; and Step-2: Assembly and brazing the samples on orbit. To prevent potential jamming during the process of mating the two halves of the double wall container and the extraction of the brazed inner container, a double cone-within-cone approach has been conceived. The results of this study are described and discussed in this manuscript.

2.0 Materials properties

In order to select effective materials for the fabrication of the container parts, a literature search was performed to identify the available properties to support the analytical modeling (**Table 1**). The selected materials were chosen for the ability to execute the process above 500ºC to enable pyrolysis sterilization by heating the seamed sections above the temperature that destroys carbon bonds and potential presence of biological content. For the base material of the containers, Stainless Steel alloy 304 was used and Silvaloy 630 (Lucas Milhaupt) was chosen for the brazing material. The latter has a 690ºC Solidus temperature (melting point) and an 800ºC Liquidus (flow point) temperature.

Table 1: Potential materials for the container and the brazing

Chamber Material	Braze Alloy	Braze Temp	Comments
Aluminum	Al-Si (4047 Al)	595C	Aluminum is dip brazed in a strong flux because of the oxide and control of the temperature is critical. Ni plating sometimes is used, but it is a technical challenge.
Copper	Cu-Ag (Cusil) Cu-Ag-P (Sil-Fos) Ag-Cu-In (Incusil 15) Au-Ge (Georo)	780C 705C 705C 361C	The oxide on Cu is weak permitting, in some instances, brazing without flux. Cusil and Sil-Fos are the most common braze alloys. Cusil or Incusil is used when phosphorus contamination must be avoided and Cusil is higher in strength. Using Georo may be advantageous because of the low melting temperature but it is not applicable for the ≥500ºC sterilization requirement.
Nickel	Cu-Ag (Cusil) Ag-Cu-In (Incusil 15) Au-Ge (Georo)	780C 705C 361C	Similar comments to Cu. However, phosphorus can be a big contamination problem when brazing Ni.
Stainless Steel	Au-Cu Au-Ni-V-Mo (Nioro ABA) Au-Ni (Nioro) Ag-Pd-Ga (Gapasil 9) Ag-Cu-Ti Cusil ABA) Ag-Cu-Pd (Palcusil 5)	970C 960C 955C 880C 815C 814C	Stainless steel is usually brazed in vacuum at high temperatures to dissolve the strong oxide. Active braze alloys may be superior. Flux may raise contamination issues, if required, as the fluxes used with stainless steel are generally fluoride and/or chloride based.
Titanium	Ti-Cu-Ni (Ticuni) Ag-Pd-Ga (Gapasil 9) Ag-Cu-Pd (Palcusil 5)	960C 880C 814C	Titanium is usually vacuum brazed with an active braze alloy due to the strong oxide. The use of plating can be a poor option.

3.0 Cylindrical container configuration concept

The development of the BTC brazing process started in 2004 and initially the focus was on cylindrical configurations that had the size of a coffee cup in which returned samples could be contained. The task objective has been to demonstrate the feasibility of using brazing as a process of synchronous sterilization, seaming, sealing, and separation. The details of the process were covered in two NASA New Technology reports [3 - 4]. A schematic diagram of the concept is shown in

Figure 1a, with a close-up in **Figure 1c**. The original design and details were developed for execution on the surface of Mars. The method is based on using a double wall container having internal gap simulating "Earth clean" volume. A brazing material seams the two containers/walls where the inner one is used for the returned sample and (upon separation of the inner container) the external one becomes integral part of a lander. The double wall container is brought with the sample to an intimate contact with the lid that, initially, is part of the lander structure. Upon heating with an inductive heater, the lid is brazed to the inner container while the outer container is brazed to the lander separating the "local-planet-dirty" environment from the "Earth-clean" section of the lander. Thus, it becomes a separator between the Earth clean zone and potential contamination from Mars. A spring at the bottom between containers is used to eject the inner container once the brazing material melts separating the two containers. The use of a brazing material that melts at temperatures that are higher than 500°C sterilizes the areas that were exposed to potential contamination (at the interface between the lid and the container). To protect the sampled material inside the inner container, a thermal insulation layer is used (**Figure 1c**). In **Figure 1c**, the red area with the rectangular cross-section shape shows the sections of the lid and the external container that enable the separation. Upon melting the brazing material, the braze flows under surface tension/capillary forces while the force from the spring drives the separation. Due to the melting: (a) the lid is brazed to the inner container and is ejected; and (b) the external wall of the container is brazed to the structure of the lander. The red circular areas represent secondary brazed joints that provide sealing and seaming.

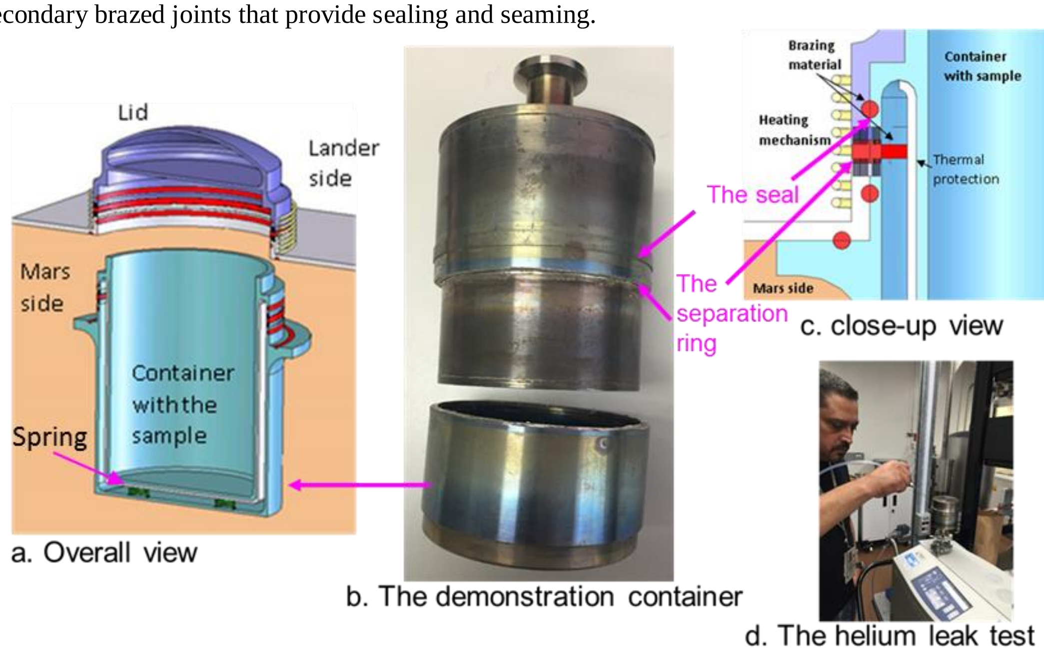

Figure 1: The cylindrical container design (a. and c.), the demonstrated breadboard (b.) and leak test (d.).

The S4 process is enabled by two sets of brazing rings including: (a) sealing/seaming rings and (b) separation ring (**Figure 2**).

a. Sealing/seaming rings - The sealing/seaming rings are produced using grooves, which are machined onto the surface of the container wall. Then, a brazing wire is inserted into the groove and once the wire is heated and melted, the excessive braze on the wall surface is shaved to leave braze filler inside the groove (**Figure 2 left**). When the S4 process is executed, two adjacent filler braze materials are aligned and upon melting them they unit to form a seam and a seal.
b. Separation ring – For the fabrication of the separation ring, the container interface that is supposed to be separated is filled with braze and melted, then it is shaved on both sides (**Figure 2right**). Upon heating and melting of the separation ring, the force from the spring lead to the separation resulting ejection of the seamed/sealed container.

The process was demonstrated in a vacuum chamber and the induction heating was done by inserting the container assembly inside the coil and connected to the drive electronics via a feed-through to the drive electronics (**Figure 3**). To minimize oxidation, once the vacuum reaches a level of milli-Torrs, research grade Argon with 99.99% purity back-fills the chamber and the vacuum pumping is resumed. A successful sealing and separation process was demonstrated and the container produced is shown in **Figure 1b**. The verification of the sealing was done using a spectrometer based He leak detector by creating a vacuum inside the container connected to the spectrometer and spraying helium on the outside next to the separated and seamed area (**Figure 1d**) [6].

During the process of heating the container, it was mounted on a support fixture using a bottom screw for aligning the brazing rings with the center plane of the coil. In addition, a retaining bar was placed above the container to keep the inner container from an uncontrolled ejection during the melting of the braze material. The assembly was heated until the brazing rings melted and created flow of the brazing material as needed to accomplish the S4 process. The process was stopped when the formed container (seamed lid and the inner container) were ejected by the force of the spring located between the inner and outer containers (**Figure 1a**).

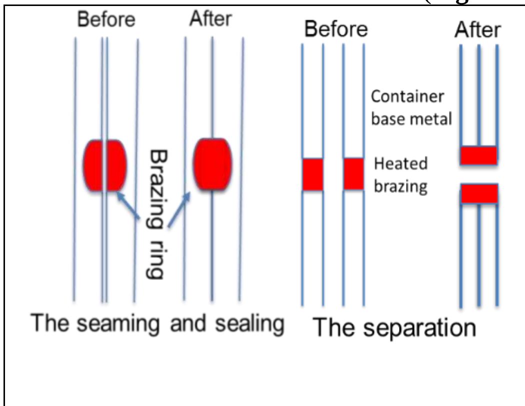

Figure 2: The seaming/sealing and separation rings

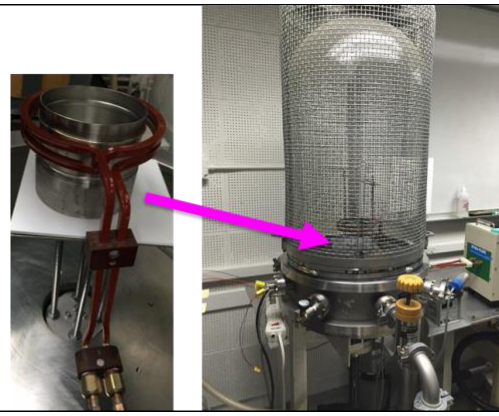

Figure 3: Left: The coil of the induction-heater is shown with a cylindrical container component. Right: The bell-jar vacuum chamber with the coil inside.

4.0 Spherical container configuration concept

As the concepts for potential Mars Sample Return have evolved, the shape of the notional container has been modified to be spherical and its size has been increased to 27 cm diameter. This container is designed to be filled with sample tubes for return and to be launched to orbit via a conceptual Mars Ascend Vehicle (MAV). This spherical container is called an Orbiting Sample (OS). The location for the execution of the BTC process has been changed from on the surface of Mars to on-orbit about at an altitude of 500 km. The OS would need to be contained and subjected to the BTC brazing S4 process and then inserted into an Earth Entry Vehicle (EEV) in an orbiting spacecraft.

The details of the proposed S4 process were enhanced to address the lessons learned from the various brazing tests and it has been designed for execution in two steps as follows:

Step-1: This Step is the hardware fabrication on Earth prior to the mission. It involves a double-wall container with brazing rings and a separation joint.

Step-2: This process is designed for execution on-orbit.

- Synchronously, the container that contains the OS is sterilized, seamed, sealed, and separated.
- The sealing is technically a welding process since it brazes pre-seal rings by melting the brazing metal surface.

As pointed out previously, the BTC-brazing sterilization is the result of heating above 500ºC for several minutes (depending on the structure and materials). Further, the sealing, seaming and separation take place synchronously:

- Unless the braze melts, the OS is not transferred to the Earth-clean area in the sealed and seamed container that is externally Earth-clean.
- Synchronously with the separation of the sealed container, the outside shell of the double-wall hemisphere "closes the door" to Mars contaminants.

The key challenge that was identified when using the cylindrical container configuration has been the possibility of jamming when inserting the double wall container into the lid. This risk has been addressed by extremely careful insertion when the process was done manually. However, this autonomous insertion is expected to be a great challenge for a robotic mechanism on-orbit. To address this jamming concern, a cone-within-cone design was conceived and a schematic illustration is shown in **Figure 4** [6]. Here, the open edge of the double wall container has an interface with a cone configuration. Similarly, the base lid has a cone configuration at its open edge. The two cone angles match each other and they are used to guide the double wall container when it is brought in contact with the lid for precision alignment. In addition, it eases the heat transfer in the process of melting the braze material. After the induction coil is activated and the brazing materials have melted, a spring between the outer and inner walls of the double wall container keeps the inner wall container preloaded against the lid. The lid position is controlled by an active element such as an actuator or a passive element with a timer. The newly formed container encloses the OS vessel (with the samples) but continues to be held in place until the joint, which is formed along the conical surface, solidifies. The separation of the container is done along a second conical shaped surface oriented opposite to the initial conical surface such that it allows for the movement of the container axially away from the coil. This increases the radial gap between the moving parts and, therefore, preventing the possibility of parts being jammed. The mechanism steps consist of the following: approach, contact, coil activation, separation, coil deactivation and cooling (**Figure 5**). The angles of the two cones and the number of the brazing rings can be chosen based on space availability and the need to use a smaller or larger volume of braze material.

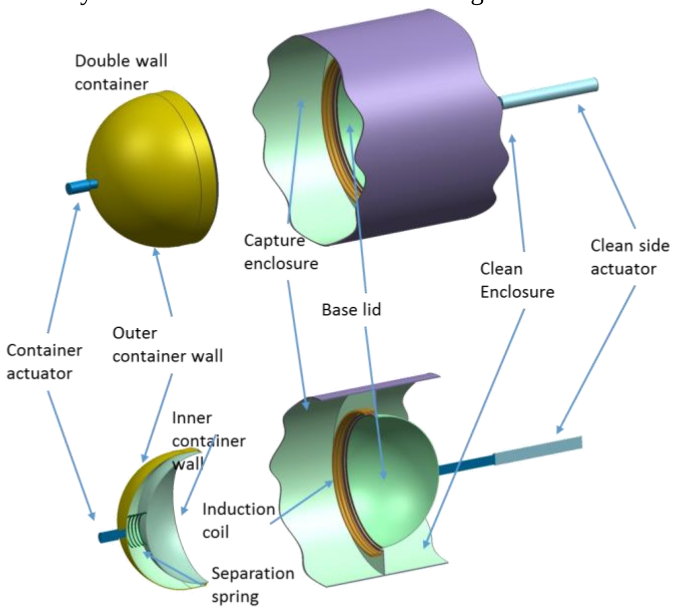

Figure 4: The components of the S4 mechanism with the cone-within-cone configuration concept.

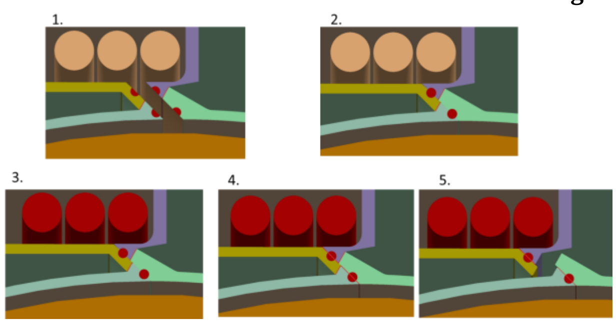

Figure 5: The S4 process steps and the double cone-within-cone representation.

The S4 mechanism can be done with directed heating and the container can be made of nonconductive material while the seal and its surrounding area are made of materials that can be easily heated with inductive heating. A practical breadboard implementation is shown in **Figure 6**. On the left, an illustration shows the spherical container, the cover merged, and the location where the separation takes place upon heating. Also shown is the location of the seaming and sealing on the cover (top three brown circles) and on the external surface of the container seaming and sealing to the external structure (bottom three brown circles). On the right of **Figure 6**, the separated container and the resulting sealing seaming sections are shown.

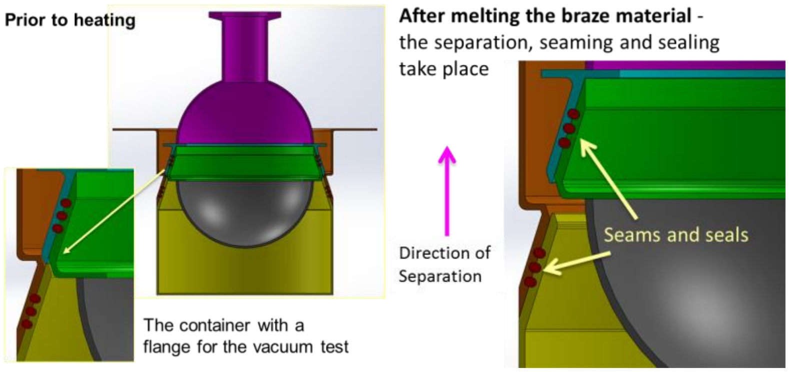

Figure 6: A breadboard implementation of the container with double cone-within-cone that is seamed, sealed, separated and sterilized before and after the activation of the heating to melt the braze material.

The process is summarized graphically in **Figure 7** with the Step-1 prepared components are shown in **Figure 7a**. The Step-2 process takes place after the capture and enclosing the OS and the activation of the induction heater to make the braze seal/seam and the separation braze rings melt. Successful demonstration of the cone-within-cone configuration has been done where a set of parts have been brazed and tested leak-free using spectrometer, vacuum and helium spray as described earlier (**Figure 8**). Even though the seal was successful, during the S4 process of brazing the prototype container that is shown in this Figure, the cover has shifted from the axis of the container. To address this issue, a registration feature was added to the conical interfaces of the lid and the container and the process was repeated. Again, the container that was subjected to the two steps process was found leak free and, in addition, the registration features produced a successful alignment during testing. Photos of the separated container and the leak test system are shown in **Figure 9**.

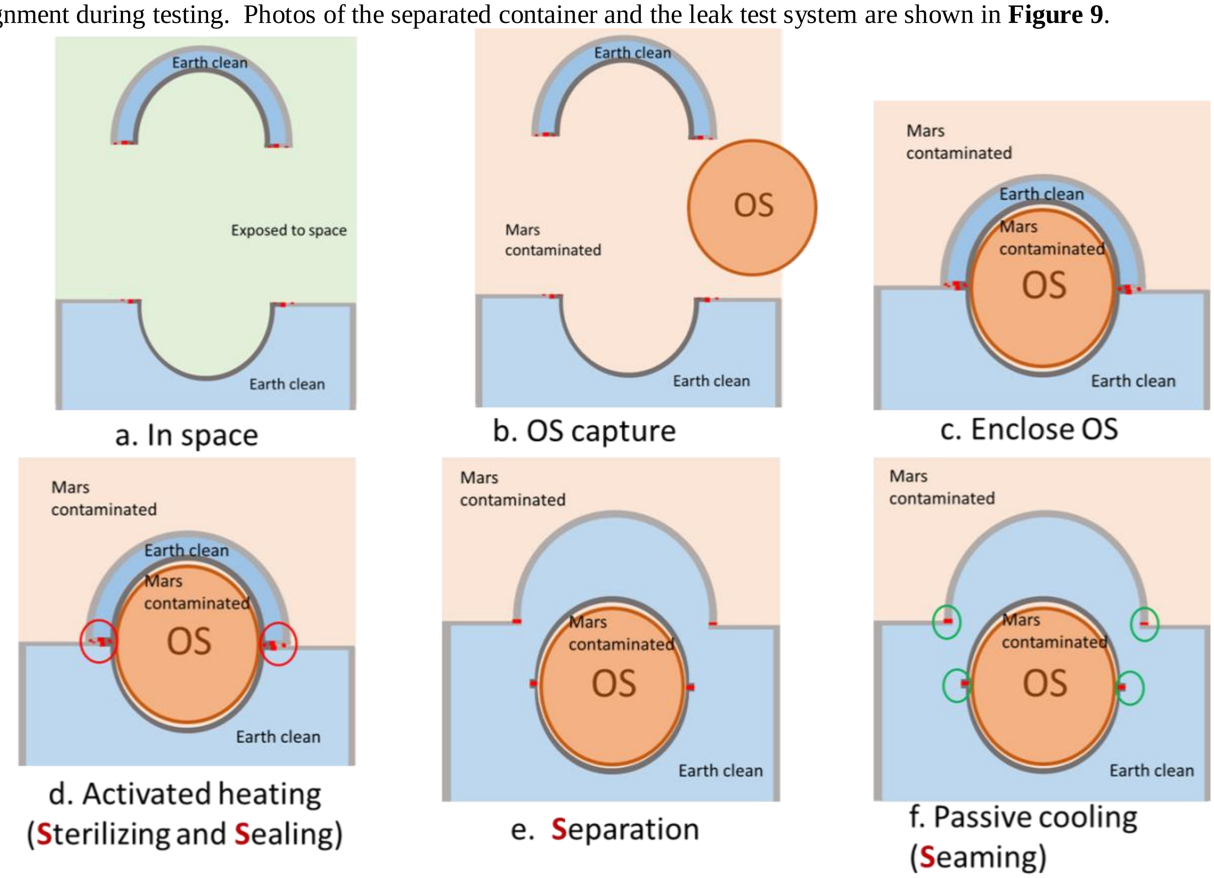

Figure 7: The Step-2 process of synchronous Sterilization, Sealing, Separation, and Seaming on-orbit

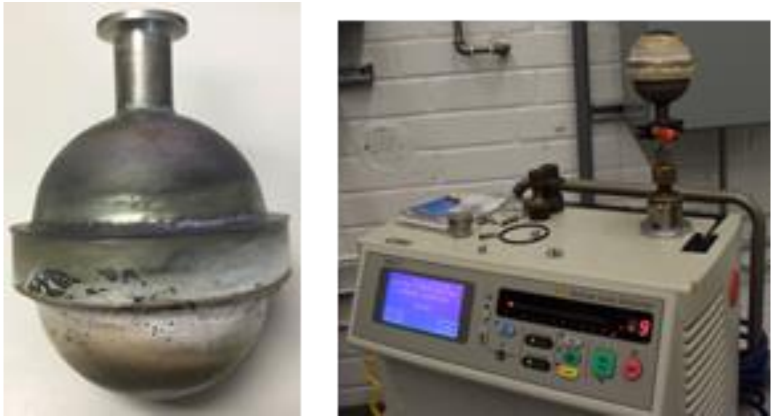

Figure 8: The produced prototype container using the full S4 process (left) and the leak test setup (right).

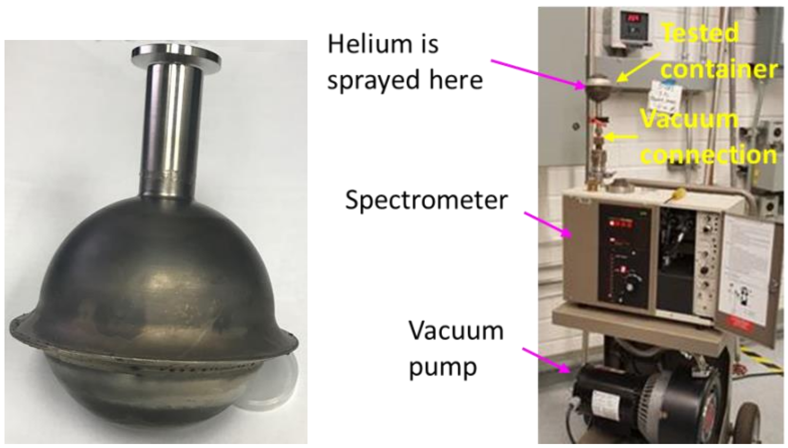

Figure 9: The container that was introduced with registration for the alignment (left) and the leak test setup (right)

5.0 Summary and conclusions

Potential Sample Return from Mars and other planetary bodies would require protection of our planet from backward contamination. For this purpose, it would be necessary to enclose the acquired samples inside a sealed container and destroy any contaminants on the external surface before returning the container to Earth. A brazing method has been developed and described in this manuscript allowing meeting the containerization and protection requirements and its feasibility has been demonstrated [3 - 6]. The initial method of sterilization and sealing has been developed using a cylindrical configuration. However, the related design did not effectively address the potential issues that are involved with putting in contact and aligning the container components and separating the container from the launch vehicle after it was seamed and sealed. A modification of the mechanism of using brazing and a double-wall container design has been developed that allows for seaming, sealing and sterilization. This separation of the container from the launch vehicle either on the surface of the explored planet, on-orbit, or on Earth can be accommodated by the technology. A double wall configuration is used with clean inner-walls allowing brazing the inner container with the returned sample. For sealing the container, the use of brazing material that melts at temperatures that are higher than 500^{o}C assures the destruction of biological materials via pyrolysis. A cone-within-cone design has been conceived and demonstrated to allow a robotic mechanism to reliably handle and manipulate the components of the process. It is significant to note that the developed BTC process based on brazing has been chosen as the primary containerization method for potential Mars Sample Return.

6.0 Acknowledgment

Research reported in this manuscript was conducted at the Jet Propulsion Laboratory (JPL), California Institute of Technology, under a contract with National Aeronautics Space Administration (NASA). The authors would like to thank Robert Gershman, JPL, for his support of the task that is results are reported in this manuscript.

7.0 References

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