

**SAMPLE TUBE SEALING FOR FUTURE PROPOSED MARS SAMPLE RETURN MISSIONS.** P. Younse, D. Aveline, X. Bao, D. Berisford, P. Bhandari, C. Budney, F. Chen, M. Cooper, S. Chung, P. DeGrosse, and D. Lewis, Jet Propulsion Laboratory, California Institute of Technology, Pasadena, California, 91109

**Introduction:** A key premise of a proposed Sample Caching Rover, a crucial element of the proposed Mars Sample Return (MSR) campaign, is that the samples could be packaged and left on Mars for an extended period of time (at least five Mars years) without loss of scientific value (Fig. 1). The MEPAG E2E-iSAG (2011) concluded that the single most important factor in preserving the scientific integrity of the samples during the interval between their collection and their analysis is effective sealing of the samples [1].

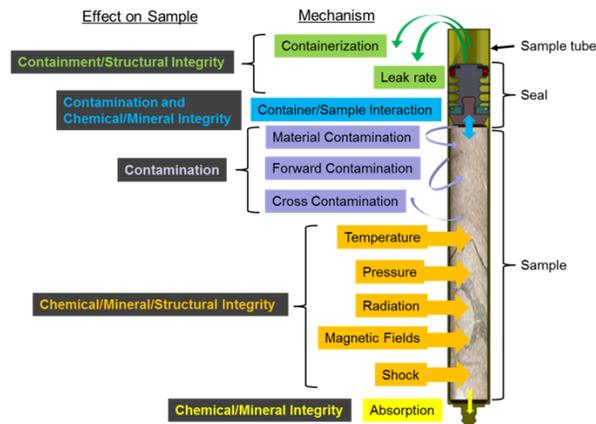


Figure 1. Various mechanisms dealing with sample integrity.

**Sample Tube Sealing Techniques:** Six sealing methods for encapsulating samples in ~1 cm diameter thin-walled sample tubes were designed and prototyped [2]. These methods include a spring energized Teflon sleeve plug, a crimped tube seal, a heat-activated shape memory alloy (SMA) plug, an SMA activated cap, a solder-based plug, and a solder-based cap (Fig. 2). Material compatibility with potential sample minerals was also studied to select proper tube and seal materials that would not lead to adverse reactions nor contaminate the sample minerals (Fig. 3).

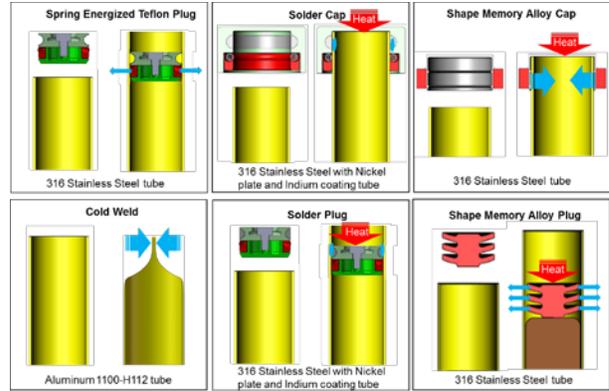


Figure 2. Seal designs.

Downselected Materials List	Ferric Sulfate $Fe_2(SO_4)_3$	Gypsum $CaSO_4 \cdot 2H_2O$	Epsomite $MgSO_4 \cdot 7H_2O$	Jarosite $KFe_3(SO_4)_2(OH)_6$	Halite NaCl	Magnesium Sulfate $MgSO_4$	Potassium Chloride KCl	Sodium Sulfate $Na_2SO_4$
302/304L CRES	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow
Al 6061-T6	Red	Red	Red	Red	Yellow	Red	Red	Red
Nitinol	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow
Teflon	Green	Green	Green	Green	Green	Green	Green	Green
Ti-6Al-4V	Yellow	Yellow	Yellow	Yellow	Yellow	Green	Yellow	Yellow
CP-70 Ti	Yellow	Yellow	Yellow	Yellow	Yellow	Green	Yellow	Yellow
Ti-5Al-25Sn	Yellow	Yellow	Yellow	Yellow	Yellow	Green	Yellow	Yellow
Al 7075-T7351	Red	Red	Red	Red	Yellow	Red	Red	Red
303 CRES	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow
Nitinol N	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow
Fused Silica	Green	Green	Green	Green	Green	Green	Green	Green

■ Low risk     ■ Medium risk     ■ High risk

Figure 3. Sample/seal material compatibility study.

**Seal Testing:** Helium leak tests were run on clean sample tubes and tubes dipped in MMS Mars simulant before and after thermal cycling between -135 C and +55 C. The crimped tube, SMA cap, and finned SMA plug seals showed hermetic seal capability compared against an industry standard of  $<1 \times 10^{-8}$  atm-cc/sec He (Fig. 4). A thermal-vacuum chamber was designed to perform future leak testing in various temperature, pressure, and atmospheric conditions (Fig. 5).

Seal Type	Initial Leak Rate (atm-cc/sec He)	Leak Rate after 1 Thermal Cycle (atm-cc/sec He)	Leak Rate after 10 Thermal Cycle (atm-cc/sec He)	Seal Strength (N)**
Spring Energized Teflon Plug – Clean	$6.3 \times 10^{-5}$	-	-	12
Spring Energized Teflon Plug – Dirty	$9.9 \times 10^{-5}$	-	-	-
Crimped Tube – Clean	$0.8 \times 10^{-10}$	$0.8 \times 10^{-10}$	$1.0 \times 10^{-10}$	408
Crimped Tube – Dirty	$8.3 \times 10^{-10}$	$0.9 \times 10^{-10}$	$1.0 \times 10^{-10}$	-
Solder Cap – Clean	$9.9 \times 10^{-5}$	-	-	173
Solder Cap – Dirty	$9.9 \times 10^{-5}$	-	-	-
Solder Plug – Clean	$9.9 \times 10^{-5}$	-	-	33
Solder Plug – Dirty	$9.9 \times 10^{-5}$	-	-	-
Shape Memory Alloy Cap – Clean	$6.7 \times 10^{-9}$	$1.8 \times 10^{-10}$	$1.0 \times 10^{-10}$	1413
Shape Memory Alloy Cap – Dirty	$2.4 \times 10^{-6}$	$0.9 \times 10^{-10}$	$1.0 \times 10^{-10}$	-
Shape Memory Alloy Plug – Clean 316 SS	$0.1 \times 10^{-10}$	$0.1 \times 10^{-10}$	$2.7 \times 10^{-7}$	1089
Shape Memory Alloy Plug – Dirty 316 SS	$0.1 \times 10^{-10}$	$0.2 \times 10^{-10}$	$4.4 \times 10^{-9}$	-
Shape Memory Alloy Plug – Clean Ti	$1.0 \times 10^{-10}$	-	$1.0 \times 10^{-10}$	2524
Shape Memory Alloy Plug – Dirty Ti	$1.0 \times 10^{-10}$	-	$4.3 \times 10^{-10}$	-

Figure 4. Sample/seal material compatibility study

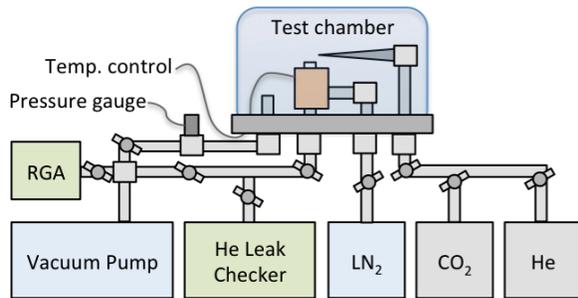


Figure 5. Thermal vacuum chamber designed for sealing and leak rate tests at various temperature and pressure.

**Analysis Tools:** Simulation tools and an IR camera testbed were developed to aid in design and analysis of the SMA-type seals (Fig. 6). Simulation models were produced in COMSOL to study the stress induced in the SMA plug and tube during swaging and sealing, as well as in ANSYS to study heat flow through the heater, plug, tube, and sample. The IR camera testbed developed provides a means to test heat-activated seals, precisely measure their activation temperatures, and qualify plugs of various geometries and alloys.

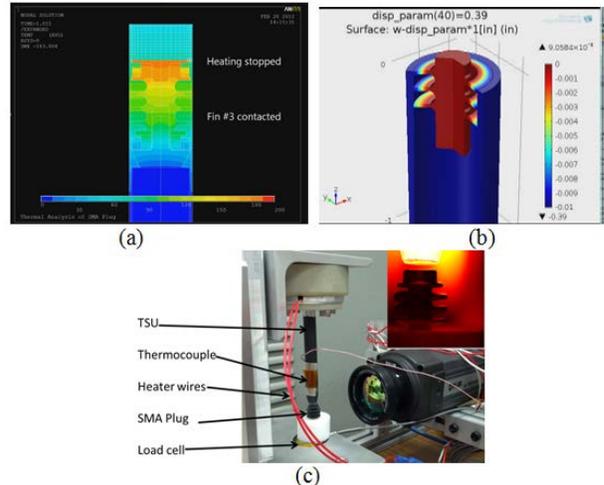


Figure 6. Analysis tools developed: (a) Thermal model, (b) stress model, (c) IR camera testbed.

**Full End-to-End Sample Integrity:** Elements spanning full end-to-end sample integrity from cleaning/sterilization before the mission to sample removal after the mission were addressed (Fig. 7). CO2 snow cleaning was tested as a potential method to both clean and sterilize material surfaces to meet future possible planetary protection/contamination control requirements, as well as offer a low temperature means to clean heat sensitive components like the SMA plugs. Seal elements were developed to allow complete integrated demonstration of sealing in a caching subsystem. A tube cutter was designed to provide a method for careful removal of the sample from a sealed tube.

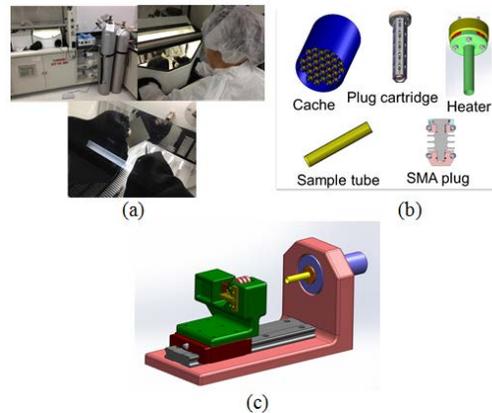


Figure 7. End-to-end sample integrity elements developed: (a) CO2 snow cleaning, (b) seal element hardware, (c) tube cutter for sample removal.

**References:** [1] MEPAG E2E-iSAG (2011) Planning for Mars Returned Sample Science: Final report of the MSR End-to-End International Science Analysis Group (E2E-iSAG). [2] Younse P. et al. (2012) IEEE Aerospace Conference, pp. 581-591.