



# Manufacturing High-Fidelity Lunar Agglutinate Simulants

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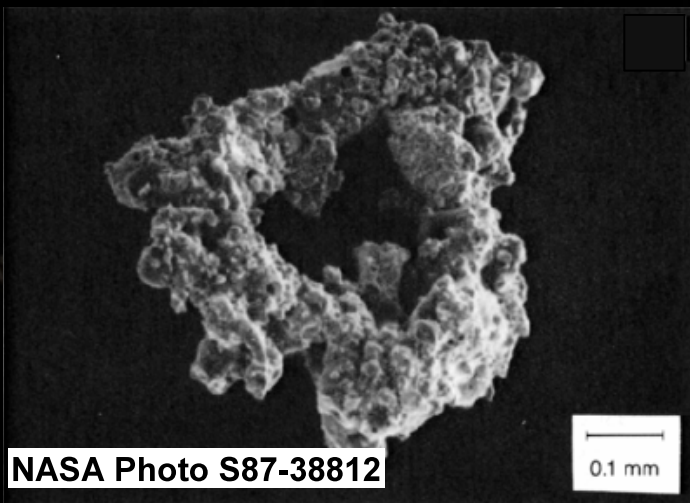
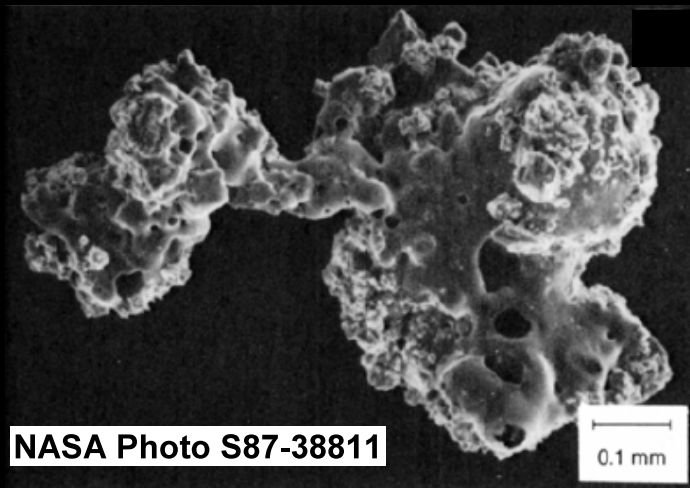
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BAE Systems/Marshall Space Flight Center

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# Lunar Regolith Properties

- The lunar regolith is very different than any naturally occurring material on Earth due to the unique, impact-dominated environment on the surface of the Moon
- Lunar regolith is composed of five basic particle types
  - Mineral fragments
  - Crystalline rock fragments
  - Breccia fragments
  - Glasses of various kinds
  - Agglutinates
- Agglutinates are abundant in the lunar regolith, especially in mature regolith where they can constitute up to 65% of the regolith by volume (Heiken et al., 1991)
- Due to the abundance of agglutinates in the lunar regolith and their unique characteristics, several organizations have been attempting to create simulated agglutinate particles

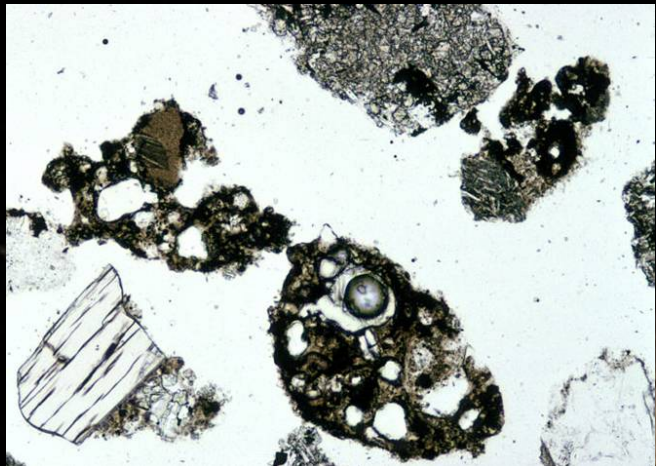
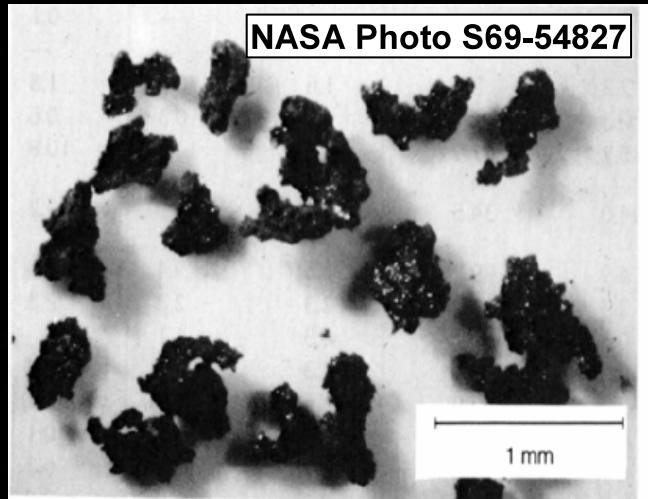
# Properties of Lunar Agglutinates



Images of lunar agglutinates

- Agglutinates are individual particles that are aggregates of smaller lunar soil particles (mineral grains, glasses, and even older agglutinates) bonded together by vesicular, flow-banded glass (Heiken et al, 1991)
  - A highly irregular shape (often with branching morphologies)
  - Heterogeneous composition (due to the presence of individual soil particles)
  - Vesicles present in the glass
  - Contains numerous metallic iron ( $\text{Fe}^0$ ) globules ranging from  $\sim 3 \text{ nm}$  –  $1 \mu\text{m}$
  - Overall size of less than a few mm

# Effects of Lunar Agglutinates

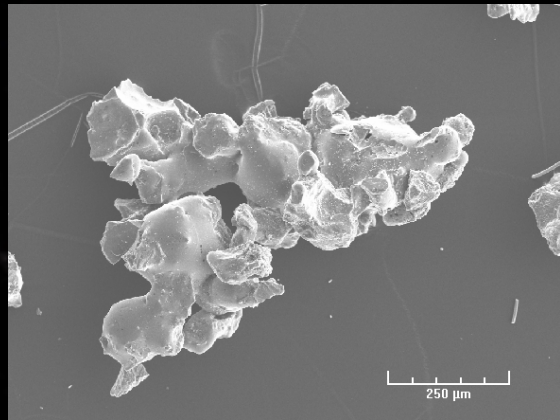


Lunar sample 68501,94 (image courtesy of Kurt Hollocher, Union College)

- The interaction of agglutinates increases the resistance shear and decreases the flowability of lunar regolith
- The mechanical properties of lunar regolith change with loading due to crushing of agglutinates
- The presence of the small metallic iron globules in the agglutinitic glass is believed by some to affect the absorption of microwave energy, the magnetic susceptibility, and the electrostatic properties of the lunar regolith



# Manufacturing Simulated Agglutinates



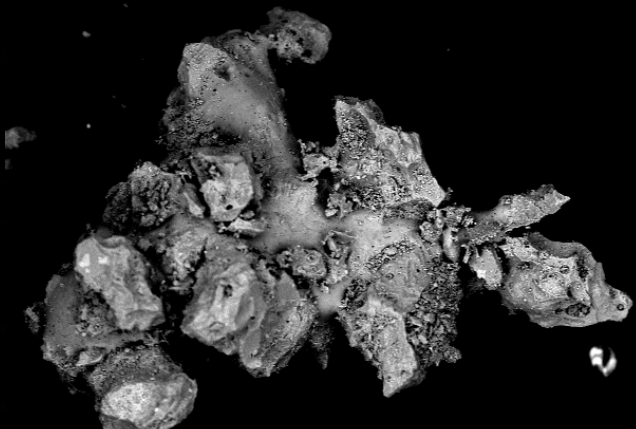
**Images of ORBITEC  
simulated agglutinates**

- Over the past 25 years, many organizations have attempted to manufacture particles that match the unique properties of lunar agglutinates
- Several groups have been successful in creating material that matches some properties of the lunar agglutinates, but development of a manufacturing process to create simulated agglutinates that match all of the primary characteristics of lunar agglutinates has been elusive
- ORBITEC, with the support of NASA, has been developing processes to manufacture high-fidelity agglutinate simulants since 2005

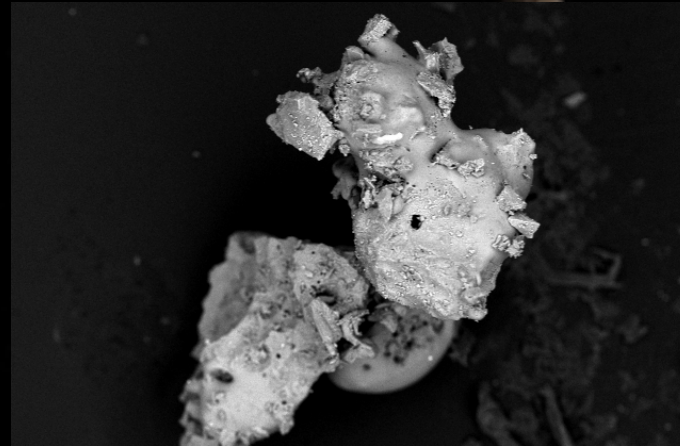
# High-Fidelity Simulated Agglutinate Requirements

- Heterogeneous composition (mixture of glass and smaller regolith particles)
- A highly irregular shape (often with branching morphologies)
- Overall size of less than a few mm
- Vesicles present in the glass
- Numerous Fe<sup>0</sup> globules ranging from ~3 nm – 1μm both on and within the glass

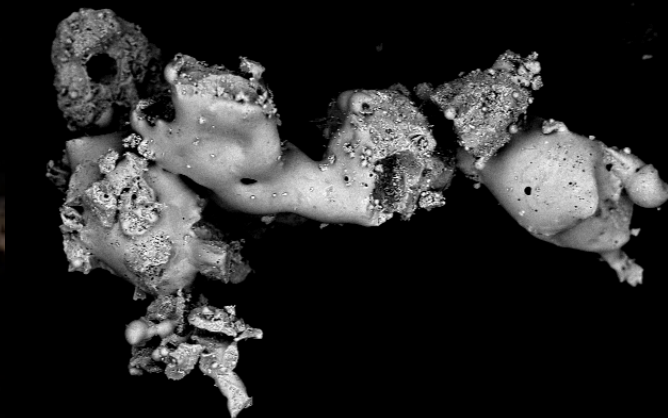
# Heterogeneous Composition of Simulated Agglutinates



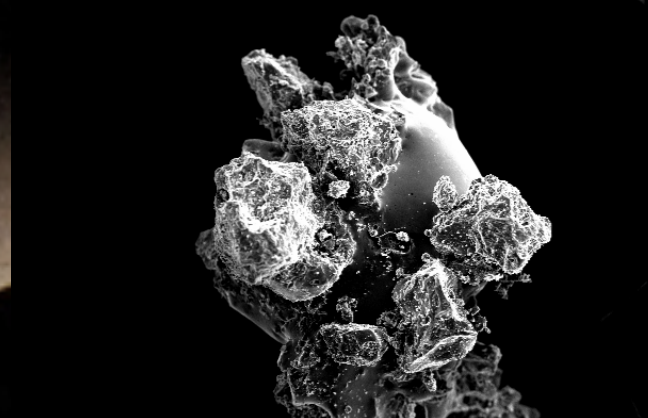
15.0kV 8.3mm x100 BSECOMP 5/14/2006 500um



15.0kV 8.2mm x140 BSECOMP 5/14/2006 400um



15.0kV 8.5mm x140 BSECOMP 5/14/2006 400um



15.0kV 8.5mm x120 SE 5/14/2006 400um

# High-Fidelity Simulated Agglutinate Requirements

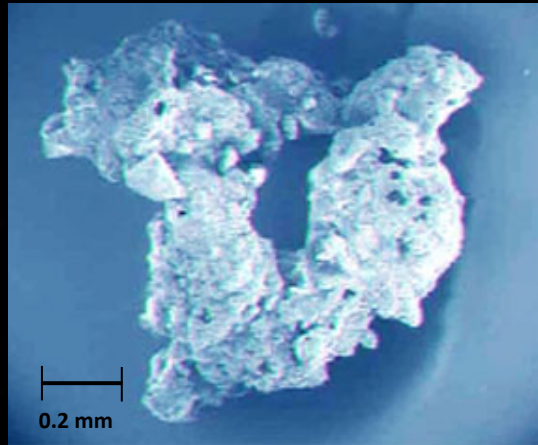


- Heterogeneous composition (mixture of glass and smaller regolith particles)
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# Comparison of the Shape and Size

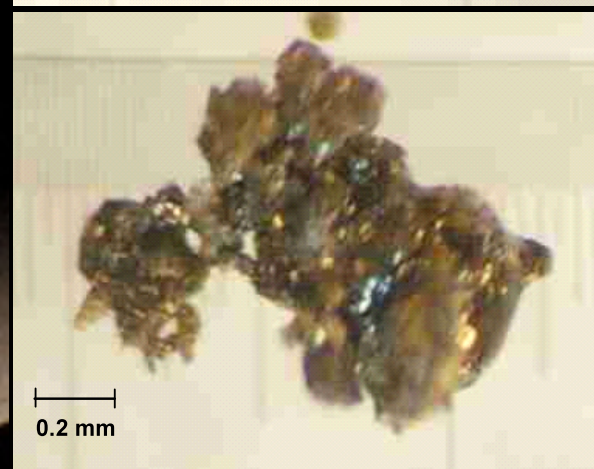
**1-mm Lunar  
Agglutinate**  
(courtesy of Dave  
McKay, NASA  
Johnson Space  
Center)



**Lunar  
Agglutinates in  
Highlands  
Regolith**  
(courtesy of Kurt  
Hollocher,  
Union College)



**ORBITEC  
Simulated  
Agglutinate  
Particles**  
(produced  
from JSC-1A  
simulant)



# High-Fidelity Simulated Agglutinate Requirements



- Heterogeneous composition (mixture of glass and smaller regolith particles)

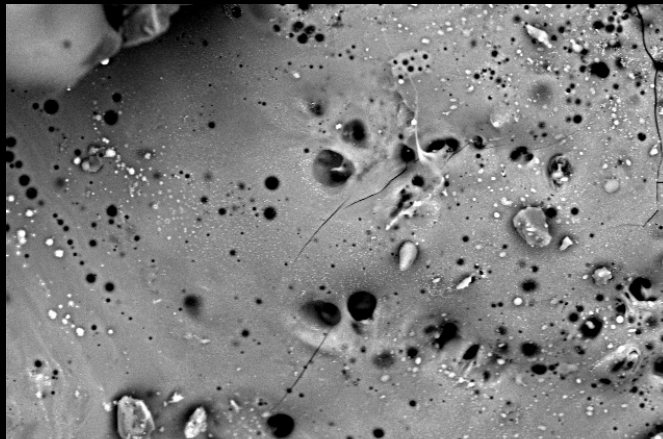


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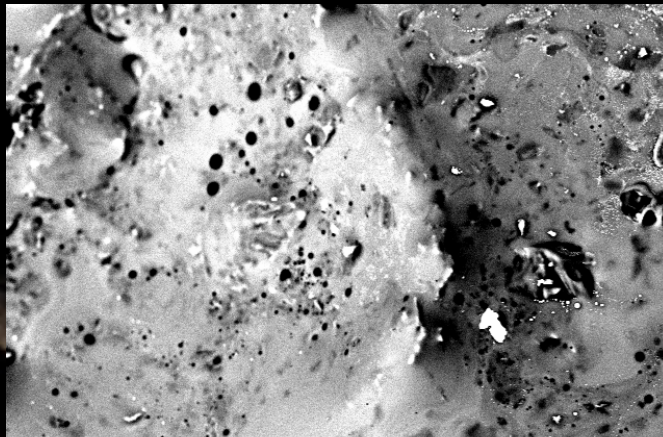
# Vesicular Glass in Simulated Agglutinates



15.0kV 8.2mm x950 BSECOMP 5/14/2006 50.0um



15.0kV 8.5mm x420 BSECOMP 5/14/2006 100um



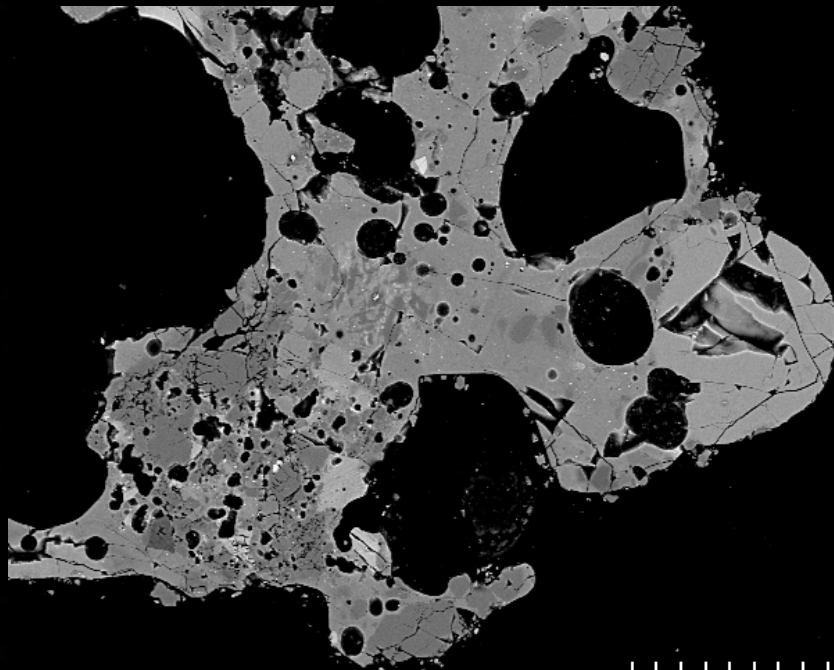
15.0kV 8.1mm x800 BSE3D 5/14/2006 50.0um



15.0kV 8.6mm x750 BSECOMP 5/14/2006 50.0um



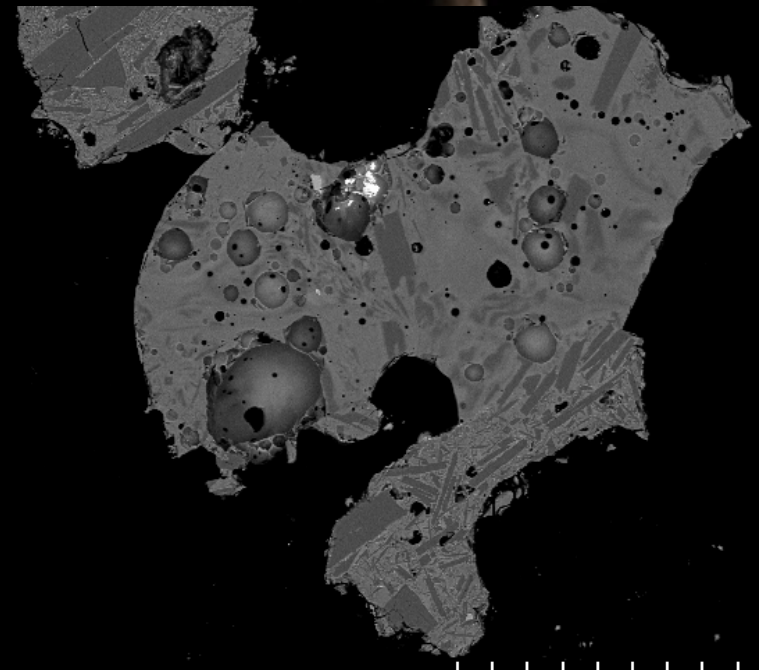
# Comparison of the Internal Structure



15.0kV x350 BSECOMP 5/19/2007

100um

**Lunar Agglutinate Thin Section  
(Apollo Sample 15103)**



15.0kV x250 BSECOMP 8/15/2006

200um

**Simulated Agglutinate Cross Section**



# High-Fidelity Simulated Agglutinate Requirements



- Heterogeneous composition (mixture of glass and smaller regolith particles)



- A highly irregular shape (often with branching morphologies)

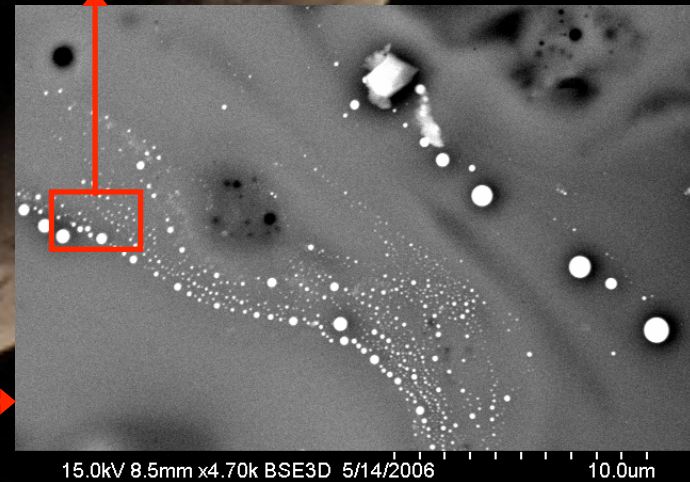
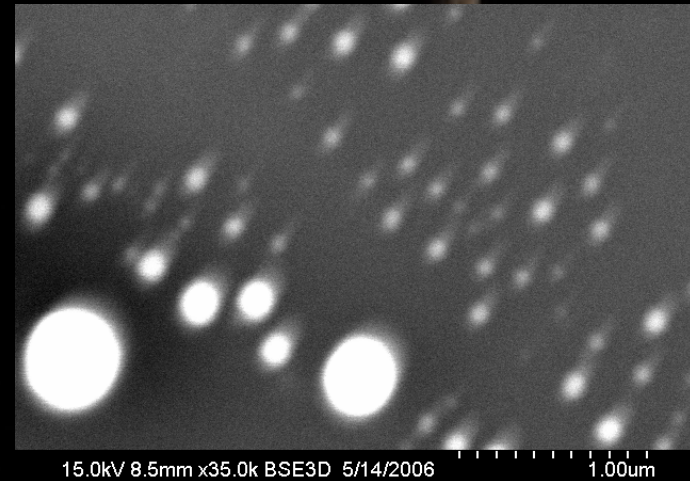
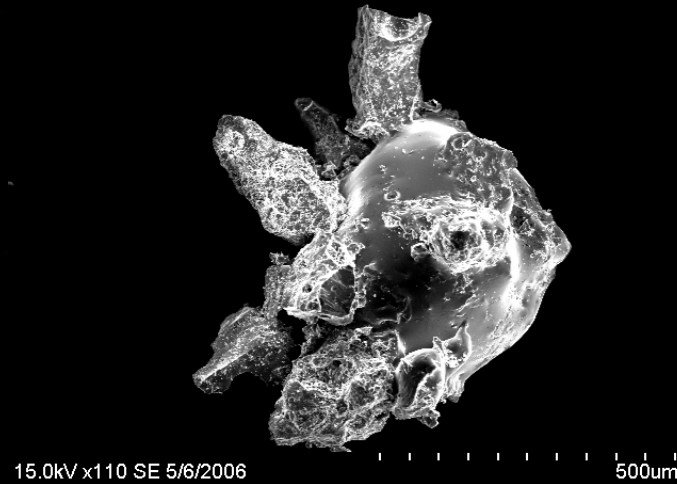


- Overall size of less than a few mm

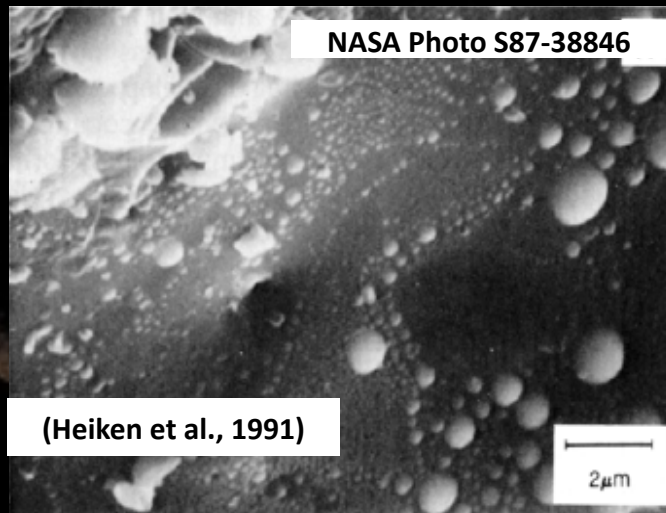
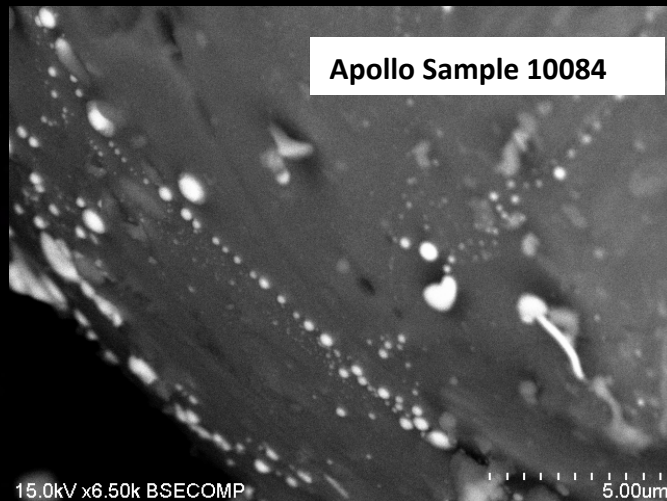


- Vesicles present in the glass
- Numerous  $\text{Fe}^0$  globules ranging from  $\sim 3$  nm –  $1\mu\text{m}$  both on and within the glass

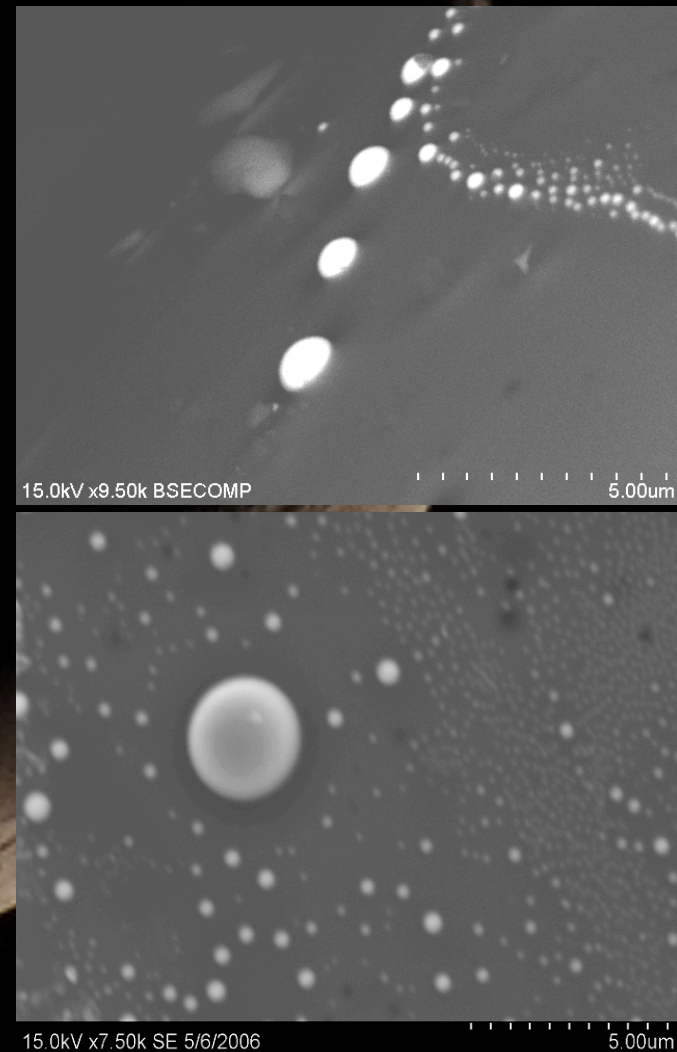
# Iron Globules on a Simulated Agglutinate



# Comparison of the Surface Iron Globules



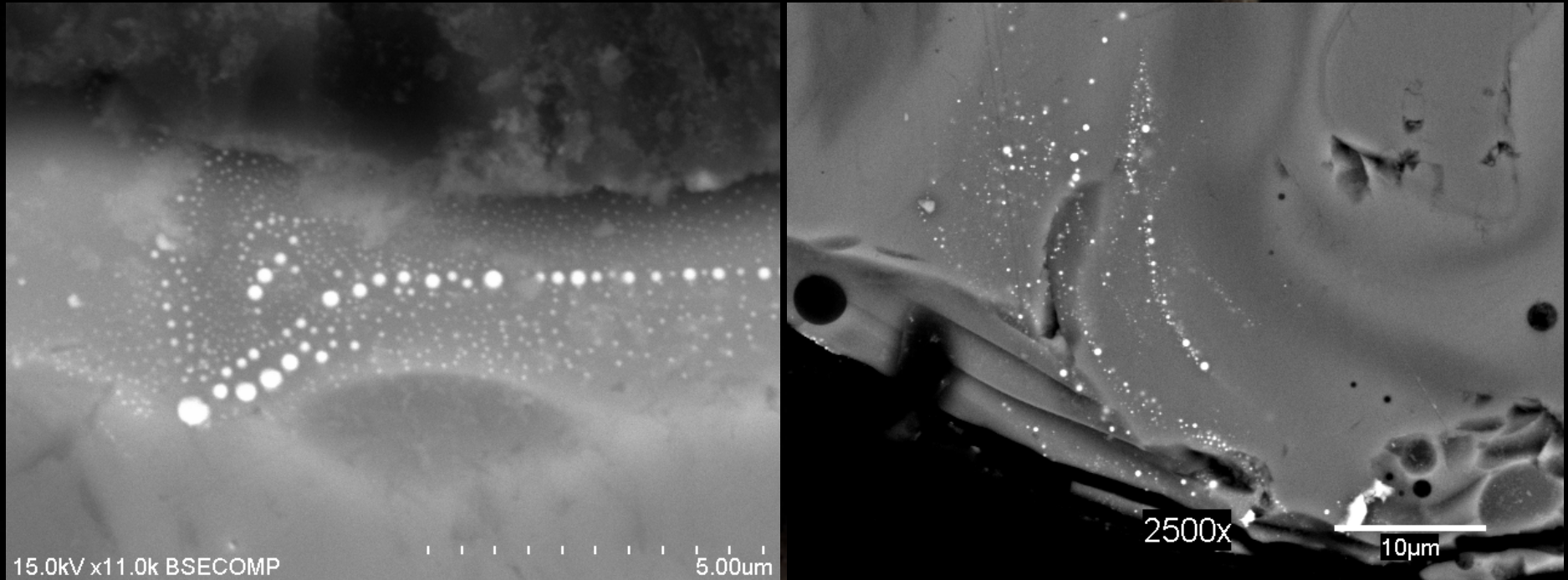
**Lunar Agglutinates**



**Simulated Agglutinates**



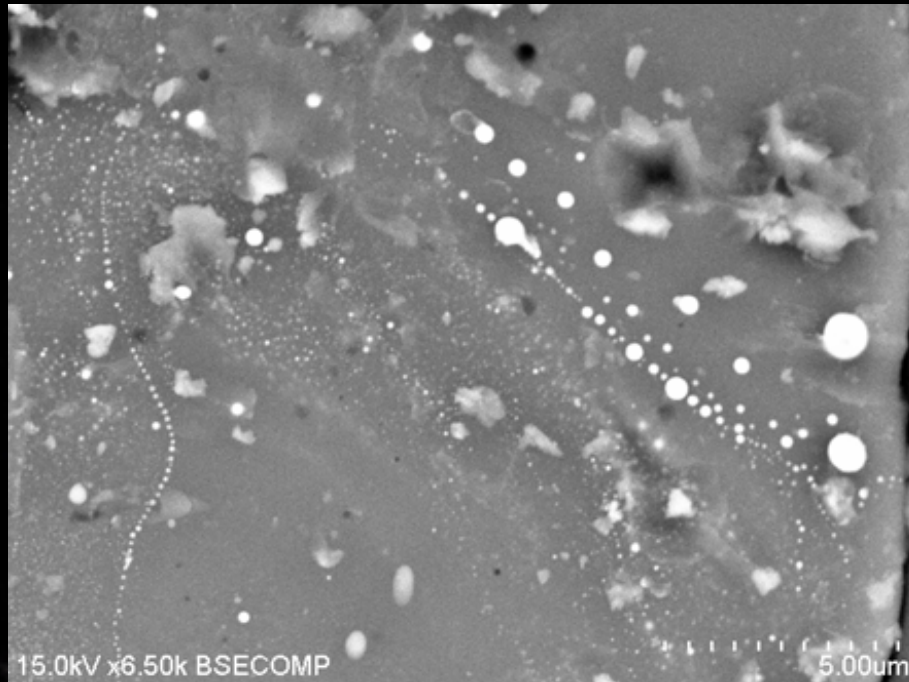
# Iron Globules Within Simulated Agglutinates



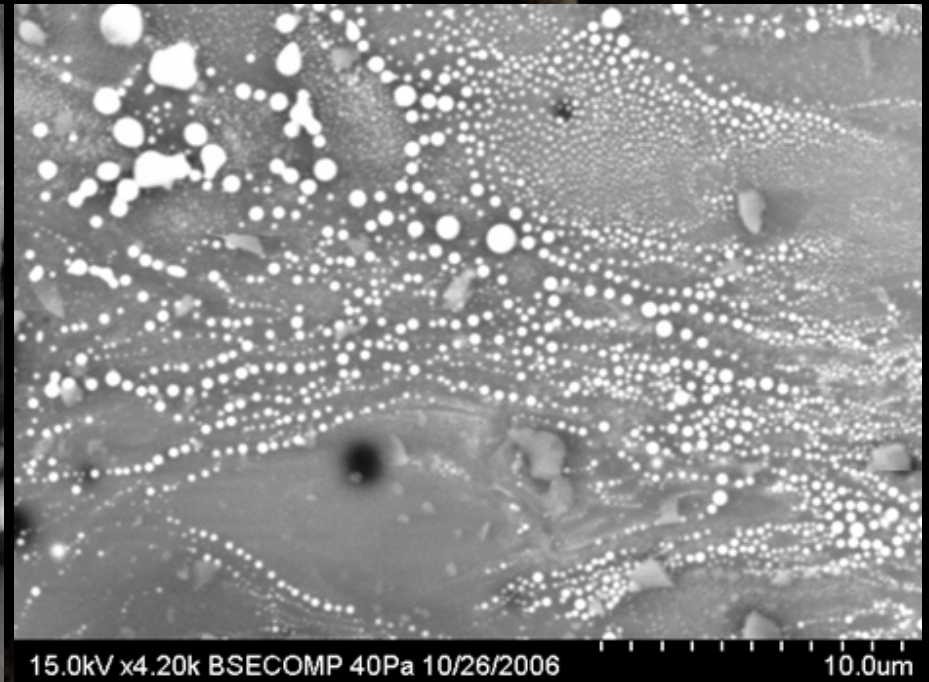
**Cross Sections of ORBITEC Simulated Agglutinates**



# Comparison of Iron Globule Size Distribution

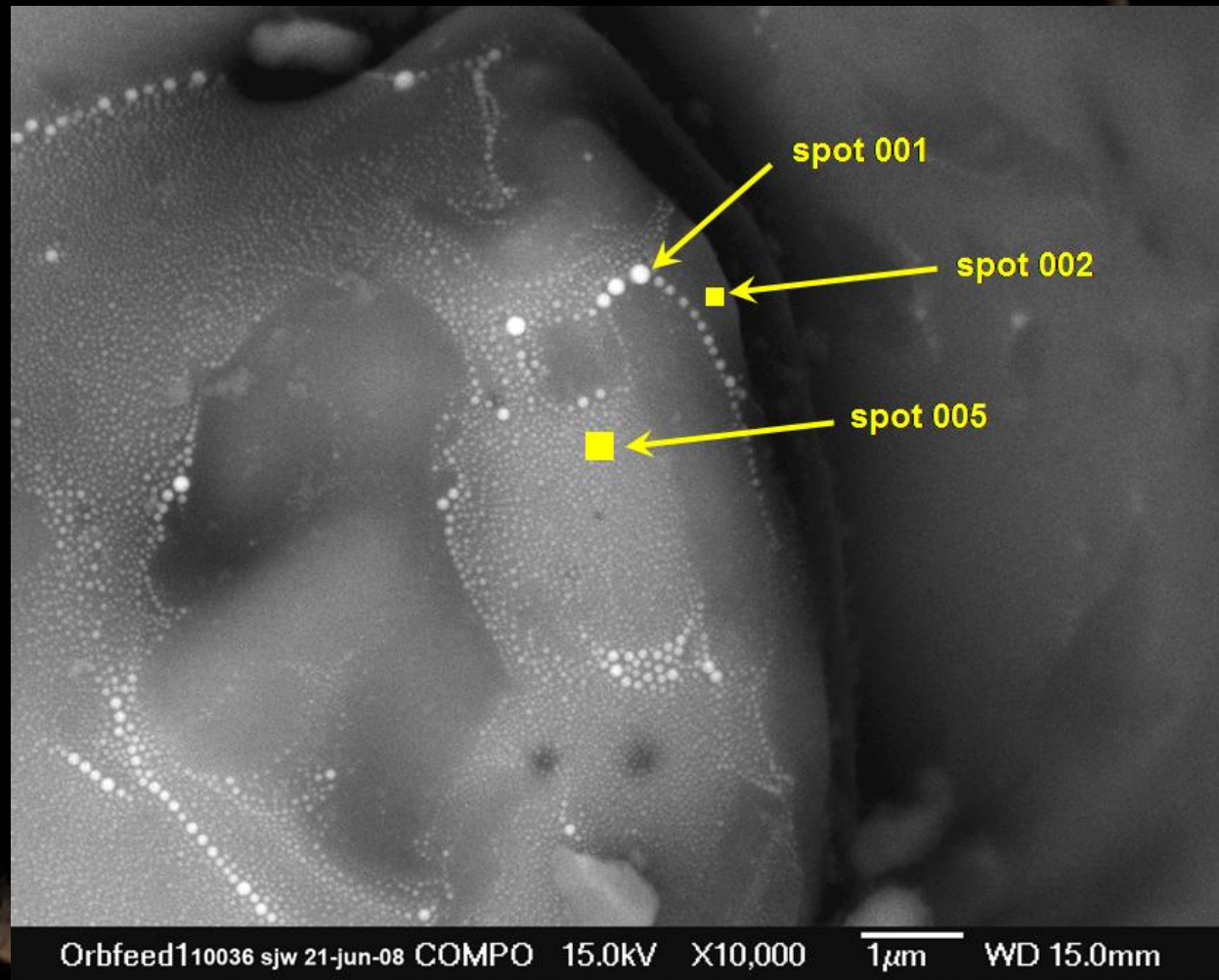


**Lunar Agglutinate Particle  
(Apollo Sample 10084)**



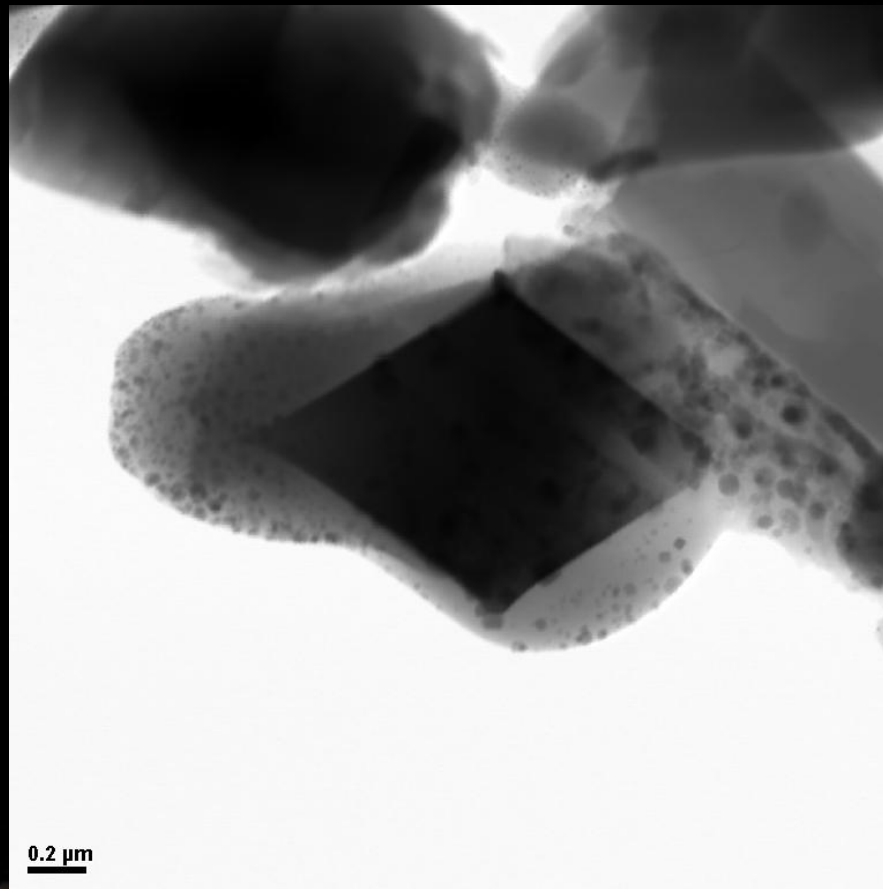
**Simulated Agglutinate Particle**

# Iron Globules on a Simulated Agglutinate



**Fe-SEM Image of the Numerous Iron Globules on a Simulated Agglutinate**  
[Image courtesy of the NASA Johnson Space Center]  
(iron globules from ~20 nm – 200 nm visible)

# Evidence of Nanophase Fe<sup>0</sup>

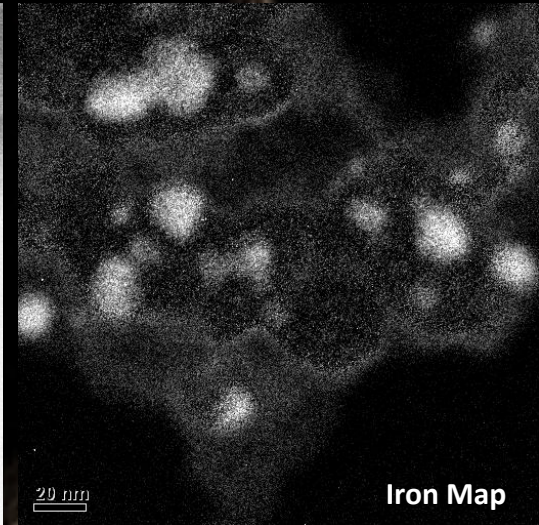
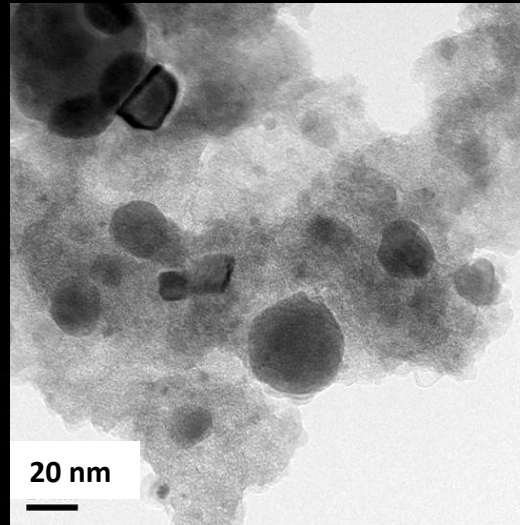
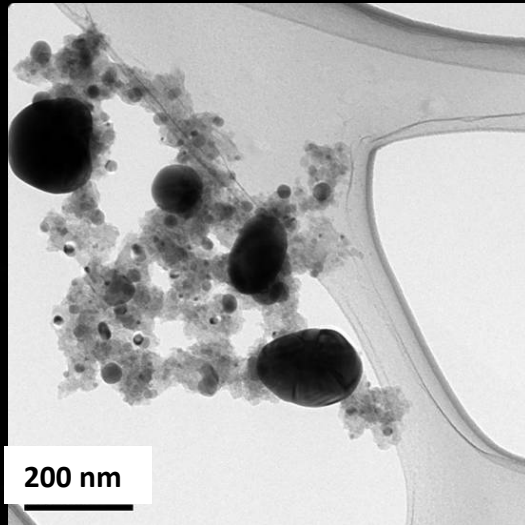


**STEM bright-field image of an euhedral Fe-oxide crystal (residual from the JSC-1A simulant) enclosed in silicate glass with nanophase globules created by the ORBITEC process. EDS analyses indicate nanophase globules are primarily Fe<sup>0</sup>.**

**[Image courtesy of the NASA Johnson Space Center]**

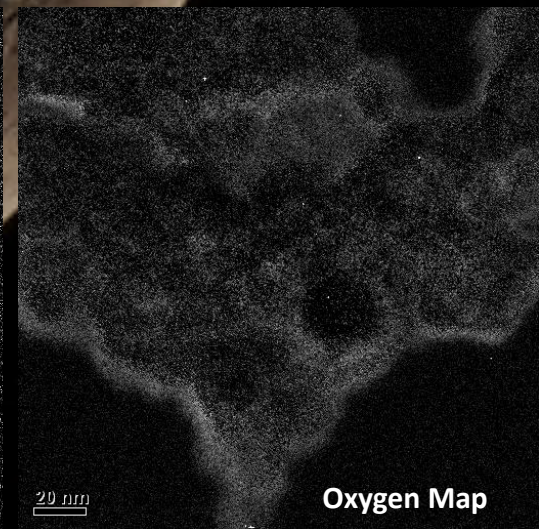
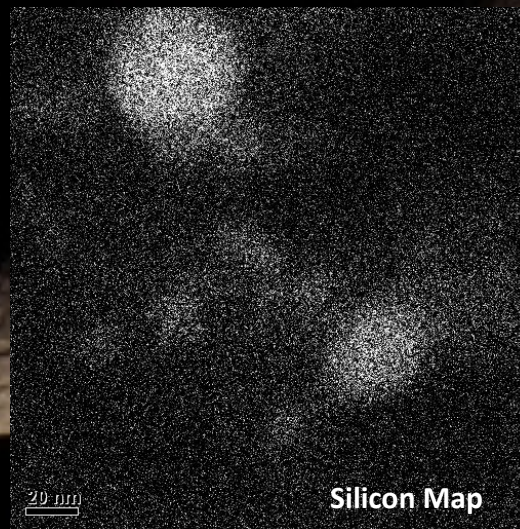


# TEM Analysis of a Simulated Agglutinate



Gatan image filter (GIF)  
maps using electron  
energy loss spectroscopy.

(Images courtesy of the NASA  
Glenn Research Center)





# Verification of the Iron Globule Composition

- Energy dispersive spectrometry (EDS) and wavelength dispersive spectrometry (WDS) performed at the University of Wisconsin-Madison indicate the larger iron globules ( $\sim 1 \mu\text{m}$ ) in both lunar agglutinates and the ORBITEC simulated agglutinates are  $\text{Fe}^0$ , not iron oxide
- Electron energy loss spectroscopy (EELS) performed by the NASA Glenn Research Center indicates that iron globules  $< 20 \text{ nm}$  do not contain any significant amounts of oxygen
- SEM and STEM analyses by the NASA Johnson Space Center indicate iron globules as small as  $5 \text{ nm}$  exist and contain primarily  $\text{Fe}^0$
- Plasma Processes Inc. recently reported an analogous process created nanophase-sized globules of  $\text{Fe}^0$  within a glass matrix as verified by EDS and electron diffraction analyses (Sen et al., 2010)

# Immiscibility of Fe<sup>0</sup> in Molten Regolith

- Metallic iron that exists in molten regolith is immiscible, so some of the globules coalesce and form larger globules
- This mechanism allows ORBITEC to produce a wide range in the sizes of iron globules like those observed in lunar agglutinates
- This mechanism also explains why all of the iron globules present in the simulated agglutinates must have the same composition (Fe<sup>0</sup>)
- ORBITEC has visually recorded the coalescence of Fe<sup>0</sup> globules in molten regolith simulant during recent carbothermal reduction experiments
- Although this is a very different process than the one used to create the simulated agglutinates, it does illustrate the behavior of Fe<sup>0</sup> in molten regolith

# Immiscibility of $\text{Fe}^0$ in Molten Regolith



# High-Fidelity Simulated Agglutinate Requirements



- Heterogeneous composition (mixture of glass and smaller regolith particles)



- A highly irregular shape (often with branching morphologies)



- Overall size of less than a few mm



- Vesicles present in the glass



- Numerous Fe<sup>0</sup> globules ranging from ~3 nm – 1μm both on and within the glass



# Conclusions

- ORBITEC, with the support of NASA, has developed a process to manufacture simulated agglutinates that match the key features of lunar agglutinates
  - Vesicular glass bonding unmelted grains
  - Size and shape typical of lunar agglutinates
  - Numerous globules of  $\text{Fe}^0$  ranging in size from  $\sim 3 \text{ nm}$  –  $1 \mu\text{m}$
- Since the finest fraction of the lunar regolith is 70-90% broken pieces of agglutinates, the ability to manufacture simulated agglutinates also allows high-fidelity lunar dust simulants to be developed

# Acknowledgements

- The work reported here was supported by NASA under the SBIR program through the Marshall Space Flight Center and the NASA Glenn Research Center
- Unless otherwise credited, the SEM images of the ORBITEC simulated agglutinates were courtesy of Dr. John Fournelle, University of Wisconsin-Madison

# References

Heiken, G., D. Vaniman, and B. French (eds). (1991) *Lunar Sourcebook- A User's Guide to the Moon*. Cambridge: Cambridge University Press.

S. Sen, D. Butts, J. O'Dell, and C. Ray. (2010) "Plasma Processing of Lunar Regolith Simulant for Oxygen and Glass Production." Earth & Space 2010, American Society of Civil Engineers, March 14-17, Honolulu, HI.