MANUFACTURING HIGH-FIDELITY LUNAR AGGLUTINATE SIMULANTS. R. J. Gustafson<sup>1</sup>, J. E. Edmunson<sup>2</sup>, and D. L. Rickman<sup>3</sup>,<sup>1</sup>Orbital Technologies Corporation (ORBITEC<sup>TM</sup>), 1212 Fourier Drive, Madison, WI 53717, <u>gustafsonr@orbitec.com</u>, <sup>2</sup>BAE Systems/Marshall Space Flight Center, National Space Science and Technology Center, 320 Sparkman Drive, VP61, Huntsville AL 35805, <u>jennifer.e.edmunson@nasa.gov</u>, <sup>3</sup>Marshall Space Flight Center, National Space Science and Technology Center, 320 Sparkman Drive, VP61, Huntsville AL 35805, <u>doug.rickman@nasa.gov</u>.

**Introduction:**The lunar regolith is very different from many naturally occurring material on Earth because it forms in the unique, impact-dominated environment of the lunar surface. Lunar regolith is composed of five basic particle types: mineral fragments, pristine crystalline rock fragments, breccia fragments, glasses of various kinds, and agglutinates (glassbonded aggregates) [1]. Agglutinates are abundant in the lunar regolith, especially in mature regoliths where they can be the dominant component. This presentation will discuss the technical feasibility of manufacturing-simulated agglutinate particles that match many of the unique properties of lunar agglutinates.

Properties of Lunar Agglutinates: Lunar agglutinates are individual particles that are aggregates of smaller lunar regolith particles (mineral grains, glasses, and even older agglutinates) bonded together by vesicular, flow-banded glass. Lunar agglutinates have many unique properties, including: (1) a highly irregular shape (often with branching morphologies), (2) presence of trapped solar wind gases (primarily hydrogen and helium) that are released when the agglutinates are crushed, (3) heterogeneous composition (due to the presence of individual soil particles), and (4) the presence of iron metal globules that are often very fine grained [1]. Figure 1 shows aback-scattered electron image of a lunar agglutinate surface. The iron globules show up as bright regions in this type of image. Approximately 99% of the iron globules in lunar agglutinates have a diameter of 1 µm or less [2] and many of them are nanophase-sized (<30 nm). The presence of nanophase-sized metallic iron (Fe<sup>0</sup>) globules are known to have a significant effect on optical properties [3] and they may enhance the absorption of microwave radiation [4].

**Properties of Simulated Agglutinates:**Several organizations, including ORBITEC, Zybek Advanced Products, and Plasma Processes Inc., have independently developed processes to produce simulated agglutinate particles with varying levels of fidelity. All of the processes create simulated agglutinate particles by partially melting a fine-grained feedstock material to produce a glass that welds together the unmelted material. Figure 2 shows the similarities between the internal and external morphologies of lunar agglutinates and the ORBITEC simulated agglutinates, including the presence of vesicles.



Figure 1. Backscattered electron image of a lunar agglutinate from Apollo Sample 10084 (left) and an ORBITEC simulated agglutinate (right).



Figure 2. (a) Lunar agglutinate [courtesy of D. McKay, NASA/JSC], (b) ORBITEC simulated agglutinate, (c) lunar agglutinate thin-section, and (d) ORBITEC simulated agglutinate cross-section.

Transmission electron microscopy (TEM) analyses performed by the NASA Glenn Research Center shows the ORBITEC process produces iron-rich globules, many with diameters <20 nm, both on and within the glassy portion of the simulated agglutinate particle, with both lunar mare and highlands compositions. These globules occur in trains and range in size from the nanometer to the micron scale, very similar to the iron globules found in lunar agglutinates (see Figure 1). In Figure 3, Gatan image filter (GIF) maps using electron energy loss spectroscopy (EELS) clearly show that the globules rich in iron do not contain oxygen.

For the larger globules (~1µm), energy dispersive spectrometry (EDS) and wavelength dispersive spectrometry (WDS) performed at the University of Wisconsin-Madison support the conclusion the globules



are nearly pure iron in both the lunar agglutinates and the simulated agglutinate particles.

Figure 3. TEM Image of a simulated agglutinate particle with GIF elemental maps.

ORBITEC can adjust process parameters to control the size of the globules. If all other process parameters are held constant, the processing time controls the maximum size of the iron globules and the resulting size distribution. Since the iron is immiscible in the melt, some of the nanophase-sized globules of iron present in the molten simulant will coalesce and form larger iron globules. ORBITEC has visually recorded this iron coalescence process at a much larger scale in carbothermal reduction experiments. In the carbothermal reduction process, lunar regolith simulant is heated to at least 1650 C in the presence of methane gas. The methane pyrolzes on the surface of the molten simulant and deposits carbon. This carbon reduces the ironoxide bearing minerals and produces sub-microscopic globules of iron. After several minutes of processing, enough of the sub-microscopic iron globules have coalesced together to form visible globules of iron. As the processing continues, some of these small visible globules continue to coalesce into larger globules often exceeding 1 mm in diameter. Figure 4 shows an example of the vitrified tephra from Mauna Kea after undergoing carbothermal reduction. Note that various sized iron globules are clearly visible in the glass. Mossbauer spectroscopy has confirmed that Fe<sup>0</sup> is produced in the processed tephra. There is no evidence that a phase change occurs as the size of the globules increases. Given that large globules are formed by the coalescence of smaller nanophase-size globules and that the large globules contain Fe<sup>0</sup>, the nanophase-sized globules observed in the ORBITEC simulated agglutinates must also contain  $Fe^{0}$ .



Figure 4. Iron globules visible in the Mauna Keatephra after carbothermal reduction processing.

The Plasma Processes Inc. process, which is analogous to the Orbitec process, produces very similar trains of high Fe content globules. These globules have been shown to be Fe<sup>0</sup> using TEM analysis [5].

**Conclusions:**ORBITEC has developed a proprietary process to manufacture simulated agglutinate particles that match many of the unique properties of lunar agglutinates. The most important feature may be the Fe<sup>0</sup> globules.The ability to manufacture accurate simulated agglutinate particles will allow higher-fidelity lunar regolith simulants to be developed. Since the finest fraction of the lunar regolith is composed of 70-90% broken pieces of agglutinates [6], the ability to manufacture simulated agglutinates ability will also support the development of high-fidelity lunar dust simulants [7].

## **Reference:**

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