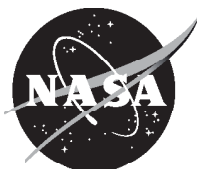


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Lunar Dust Chemical, Electrical, and Mechanical Reactivity: Simulation and Characterization

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Summary

Lunar dust is recognized to be a highly reactive material in its native state. Many, if not all Constellation systems will be affected by its adhesion, abrasion, and reactivity. A critical requirement to develop successful strategies for dealing with lunar dust and designing tolerant systems will be to produce similar material for ground-based testing.

Introduction

During the Apollo program, lunar regolith, especially the fine fraction referred to as dust, plagued astronauts on the surface (refs. 1 and 2). Effects included vision obscuration, equipment clogs, dust coating and abrading, radiator performance degradation, seal failure, and dust inhalation. Under the Exploration Technology Development Program (ETDP), several programs are developing new technologies that will enable the National Aeronautics and Space Administration (NASA) to conduct future human exploration missions. One such project is dust management, which involves the implementation of lunar dust risk reduction activities across the Agency. Developing system requirements associated with dust exposure including characterizing dust exposure, understanding the effects of dust exposure to humans and equipment, and identifying technologies can help mitigate the effects of lunar dust on surface missions. An important aspect of this technology development will be to test the possible dust mitigation solutions in an accurately simulated lunar environment on Earth (ref. 3). This will require the development of a high-fidelity simulant, possessing physical and chemical properties analogous to those of actual lunar regolith. JSC1-a has been developed to simulate the physical properties. The simulation of chemical properties will require (chemical) activation of JSC1-a or any other simulant (ref. 4) This report addresses methods of activation and characterization.

Background

Lunar regolith is considered to be activated through the processes of solar wind implantation, gamma, x-ray, and ultraviolet radiation, and micrometeorite impact. Collectively, these processes are known as space weathering (ref. 5).

Reactivity as a chemical concept has several disparate meanings. In this report, chemical reactivity is understood as the tendency for chemical bond formation upon exposure to common gases, solids, and liquids. As this reactivity may be expressed in many ways such as adhesion, abrasion, chemical reaction, and electrical conductivity, and coupled with the variety of methods by which it may be introduced—ball milling, plasma, and carbothermal reduction—chemical reactivity will be more generally referred to as activity.

General challenges in lunar regolith simulant activation are method of activation, technique for characterization, and the implementation and/or compatibility of characterization method within the

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activation environment. While the first two challenges may appear arbitrary, the choice is critical depending upon the desired properties of interest. The third represents logistical difficulties with handling and transfer operations of an active material from one unit, device, or chamber to another or with establishing suitable feedthrough connections with reliable material contact.

Methods of Activation

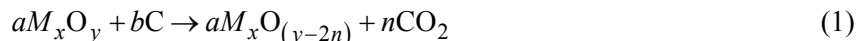
Three methods by which lunar simulant can be activated are

- (1) Chemical activation—carbothermal reduction
- (2) Electrical activation—plasma treatment
- (3) Mechanical activation—ball milling

Each of these approaches will be discussed, with advantages and potential limitations presented in each case. The degree of uniformity of activation (e.g., creation of a specific type of radical or atomic vacancy) will decrease with increasing material physical and chemical heterogeneity. Each method of activation has precedence in different fields.

Ball milling is well known to increase material surface area and fracture crystalline materials (ref. 6). It is widely used to increase surface area, pulverize particles, and break up agglomerates. It is capable of producing solid-state solutions as powders at the near-molecular level. Such physical attrition will create surface vacancies and radical sites upon particle surfaces. If conducted within an inert environment, these sites will remain unreacted and present reaction sites.

Carbothermal reduction is a well-known industrial process for nitride and carbide production (ref. 7). On a laboratory scale, the process is widely used in material synthesis processes. At elevated temperatures, the general reaction is represented by



where C is carbon, *M* represents the metal, and O stands for oxygen. Elevated temperatures are required for initiation, given the high activation energies involved.

Plasmas are well known for altering surface chemistries (ref. 8). For example, corona plasmas are presently used in treating plastics to impart hydrophilic properties. In such processes the energetic species (metastable electronically excited species, radicals, single atoms, and ultraviolet (UV) photons) rupture C–C and C–H bonds, creating radical sites by atom removal and dangling bonds by bond scission. These sites will react with water vapor to form hydroxyl groups. More generally there are many plasma types, depending upon input energy, alternating current (AC) or direct current (DC) operation, frequency of input power, electrode geometry, and gas environment of operation. Well-known plasma types include arc, glow discharge, microwave, dielectric barrier, and corona (refs. 9 and 10). These types are not mutually exclusive, and many varieties exist for each category.

Activation Methods and Characterization: Detailed Approaches

Electrical Activation—Plasma Processing

In a corona or glow plasma within an inert gas environment (Ar or He), metastable atoms, energetic electrons, and ultraviolet (UV) photons will impact lunar simulant (ref. 11). The simulant, as a thin layer, will be supported upon an interdigitated electrode (comb) pattern to facilitate electrical resistance measurements. A corona plasma, configured as a point-to-plane will create the energetic species. These species are anticipated to create atomic vacancies, remove surface adsorbates, and create dangling bonds. The combined effect will be an increase in electrical conductivity as will be measured across the

interdigitated electrodes. Comparison of the conductivity before and after plasma treatment will provide a quantified measure of activation.

The basis for this approach is the concept of the resistance change of metal oxide semiconductors upon chemisorption of oxidizing or reducing gases. Here, the plasma is used instead to create a net change in the conductivity. This change will be quantified per unit mass of simulant as deposited across the measurement unit. Plasma-processing parameters include time, energy, and electrode surface area and geometry. For the initial corona configuration, polarity also is a variable.

Logical advancements include other plasmas (e.g., hollow cathode or dielectric barrier), using a heated electrode platform, and finally a field-effect transistor (FET) configuration. A hollow cathode could be fabricated to work as a microflowing discharge. Such a plasma would provide spatial uniformity to ensure dust activation across the entire electrode assembly, irrespective of electrode pattern. If a more energetic plasma were desired, a dielectric barrier discharge (DEB) would be appropriate. In its traditional form, this plasma produces a myriad of short-lived miniature arcs between a dielectric and opposing metal electrode. Recent advances have demonstrated the potential to create a glow plasma in a DEB configuration (refs. 9 and 10). As for the corona plasma, operating parameters would require definition based on the degree and type of activation created. To measure activation, a heated electrode configuration would facilitate the electrical conductivity measurement. Wide-bandgap semiconductors, such as TiO₂, exhibit a large change in conductivity with temperature. Higher temperatures aid charge carrier mobility and hence bring the resistance to a reasonable level for measurement. Finally, an FET configuration would permit resolution of the increase in conductivity because of either an increase in charge carrier mobility or a net increase in charge carrier concentration because overall conductivity σ is expressed by

$$\sigma = enu \quad (2)$$

where e is the unit of elementary charge; n , the number of charge carries; and u , their mobility.

A particular advantage of the plasma approach presented here is the optical accessibility. Raman spectroscopy is a well-practiced method for assessing the crystallinity of material. Fluorescence is well-practiced for characterizing electronic states that may arise from interstitials, vacancies, or deep-level traps. Related, a near monolayer of dye could be deposited upon the activated material, and changes in fluorescence could be used to characterize interactions.

Chemical Activation—Carbothermal Reduction

Carbothermal reduction is a thermally activated process for reducing oxides to extract elemental metal (refs. 12 and 13). If the amount of carbon used is less than the stoichiometric quantity, the result will be a partially reduced oxide.

With the products being CO₂ and possibly CO, oxygen surface vacancies are clearly created. Corresponding to the change in stoichiometry, the metal oxidation state will concurrently change. The activated material will seek to renew these sites by chemisorption of oxygen. Both the weight loss and reactive uptake can be quantified gravimetrically. The specific procedure for performing this operation is as follows:

- (1) Mix M1300 (Cabot Corporation) or arc soot with simulant using mortar and pestle
- (2) Use 0.5, then 1.0 and 5.0 wt% of carbon to oxide simulant
- (3) Use whatever appears a convenient amount of simulant (e.g., 1 g)
- (4) Perform thermogravimetric analysis (TGA), purge well
- (5) Heat to ~1000 °C using temperature ramp, under inert atmosphere
- (6) Cool to room temperature, under inert atmosphere
- (7) Switch to air and measure the weight change as a function of a moderate to slow temperature ramp (e.g., <20 °C/min, perhaps 10 °C)

(8) Ramp temperature up to a maximum 500 °C, as any residual carbon will oxidize significantly and invalidate weight changes

The reduction step is analogous to a temperature-programmed reduction (TPR) as often employed in catalyst characterization for oxygen storage capacity (ref. 14). For a mixed oxide intimately mixed with carbon, different oxygen sites in different materials will possess different activation energies for reaction with carbon and hence, require different temperatures. The difference in the process here is that the reducing agent is not H₂ within an inert carrier but rather, solid carbon. The second difference is that reduction is not performed to completion, but only partially, to impart reactivity to the material while largely retaining its physical and chemical composition. Also, the weight loss is convolved with the carbon mass loss. Therein, the reduction step here is used as a means for activation rather than as a quantified measure of imparted reactivity.

The oxidation step corresponding to step 7 above is analogous to temperature-programmed oxidation (TPO), also used to characterize catalysts for active metal content (ref. 14). Differences here are characterization of the oxide rather than metal catalyst particles. The sample weight gain through oxidation and the corresponding temperatures for incremental increases are anticipated to provide quantified measures of simulant reactivity and reactive site heterogeneity as revealed by reactive uptake at different temperatures.

Although different sites are likely to require different temperatures for the prior carbothermal reduction, oxygen vacancies once created are likely to be sufficiently reactive that all O₂ chemisorption occurs at ambient temperature. Further resolution of the particular compounds responsible for the observed reactivity may be achieved by using pure components of the specific minerals, such as ilmenite.

Fundamental to achieving an accurate oxygen uptake during TPO is for the prior reduction process to have fully consumed the carbon, a limiting reagent. Residual carbon will oxidize at temperatures as low as ~500 °C and counter the measured sample weight gain as it gasifies. Full reduction will be aided by intimate mixing between the oxide and carbon, utilizing a carbon with high reactivity (low onset temperature of oxidation) and performing the reduction to temperatures of ~1000 °C to ensure consumption of carbon by lattice oxygen atoms. Weight loss during reduction should be proportional to carbon loss, as should weight gain. Apart from providing different degrees of partial reduction (or activation), different initial amounts of carbon will provide a further check of proportional weight losses and gains. To be reemphasized, intimate mixing of the carbon and oxide simulant is required for success.

Given these issues, clearly H₂ is a more straightforward reducing agent, for characterization purposes and lab-scale production. (To be noted, at concentrations of <4 percent, H₂-air mixtures are not flammable.) With carbon as reductant, residual carbon can complicate the subsequent measure of activation, particularly if oxide formation requires temperatures of ~400 to 600 °C. With H₂, the material may straightforwardly be reduced in stages. Incremental measures of activation may then be quantified by reactive uptake of O₂ measured gravimetrically. Activation and loss may be tied to different simulant additives and/or particular mineral phases. Differentiation of a single-step Arrhenius expression may be used to determine an activation energy. A key requirement is that the TGA balance be gas-tight, to provide accurate determination of activation, either measured by sample weight loss or gain. Operationally, it is important that reduction ramps in temperature be modest, so as to not succumb to mass transport limitations within the powder sample in the sample pan.

Mechanical Activation—Ball Milling

There are many types of ball milling that may be classified either by method or process (ref. 15). Low-energy ball milling is capable of achieving similar results as nominal high-energy milling, dependent upon the container, balls, and so forth. The appendix further summarizes the different types of milling.

So-called “mixing bowls” are available that permit milling under an inert atmosphere. What in essence are tire pressure valves are used for gas introduction and venting. Additionally, these bowls are sealed by a flat gasket. Coupling to such a system along with dead-volumes and such is seen to preclude

accurate measurement of O₂ uptake subsequent to milling. Separate ports for gas introduction and measurement would be added.

The basic physical action of milling will break or fracture particles, thereby increasing surface area and creating numerous dangling bonds or radical sites and atomic vacancies (ref. 16). Seeking to complete unfilled bonding configurations, such sites are highly reactive and are responsible for the material's chemical reactivity. For the materials TiO₂, SiO₂, and Al₂O₃, active sites will react with water to create surface hydroxyl groups. To measure such groups, the surface uptake will be measured by changes in the gas-phase water concentration. Introduction of a reactant gas, maintaining near-constant pressure in the bowl, and minimizing dead volumes in associated plumbing are obstacles to in situ measurement of the milled material. However, in this in situ approach, the advantage of minimizing sample handling post-milling avoids other complications.

After completion of milling, a measured amount of gas containing a controlled level of humidity will be introduced into the mixing bowl that still contains the milled simulant. After equilibration, a gas sample will be withdrawn and its contents analyzed by gas chromatography. The O₂ concentration changes will be quantified by reference to the change expected from dilution (into the nominal bowl volume) and by subtraction of O₂ possibly evolved during milling. This concentration would be measured by gas sampling of the head space immediately after milling. Gas introduction and extraction will be through a septum held within added ports. Presently, a calibration gas sampling connector is anticipated to serve as the septum holder, as designed. This procedure also necessitates the use of a stainless steel or carbide mixing bowl to minimize reactive O₂ uptake by the bowl's inside surface.

The present plan minimizes sample handling prior to measurement. Other characterization methods including electrical and optical testing will likely require removal of the activated material. Such transfer operations can be performed within a very high quality glovebox. Ideally, O₂ and H₂O concentrations would be on the order of 1 ppm. If such a system were available, reactive uptake measured gravimetrically could be performed using a commercial gas adsorption instrument.

Apart from process variables such as milling time, method, and the milling material, possible other considerations include the use of other reactive gases, such as hydrocarbons. Variations of the reactant can provide different probes by which surface sites of varying reactivity can be identified and quantified.

A final consideration is that additives, such as nanophase Fe, can be added to simulants prior to milling. Ball milling is a well-practiced method for creating intimately mixed materials of varied composition not readily produced by standard chemical approaches. Ball milling could be exploited not only for simulant production and concurrent activation of the same.

Scalability

Scalability is a critical issue for realistic testing of future lunar systems. With recognition that different systems will be affected by disparate aspects of lunar dust activity, each method must be assessed for its volumetric material capacity.

Ball milling is performed commercially (ref. 17). Utilizing industrial machines, hundreds of kilogram quantities may be obtained. Obviously, the transfer and dispersal of such material to a testing facility will require detailed logistical planning. An alternative is a jet mill using an inert gas. The smaller units are capable of fracturing materials with subsequent collection within a bag filter. Without the bag filter, such a unit could concurrently disperse activated material within an enclosure. After the dust had settled, pump-down could be used for vacuum operation. With suitable selection, plasma processing can also be scaled. In the absence of known commercial operation, a unit would need to be designed and built. Nevertheless, the dielectric barrier approach is used commercially for treating gaseous pollutants (ref. 18). A similar design for treating lunar simulant on a conveyor belt would be envisioned. Alternatively, dust could be dispersed and distributed within a test facility and then treated to a flowing afterglow as produced by a hollow cathode plasma operating as a gas jet (ref. 19). Carbothermal reduction is performed commercially (refs. 20 and 21). With a known recipe for the weight percent of carbon and temperature program, in-house kilogram scales could be easily realized using commercial furnaces. If a

large tube is used, a holder is compatible with the majority of moderate scale furnaces, coupling to a test chamber would be straightforward. Large quantities would require design and modification of industrial systems to maintain the material under an inert environment.

Future Directions and Efforts

Future efforts under the ETDP program should focus upon cross comparison between measures of reactivity with an activation method. Oxygen uptake will be compared for all three methods of activation but may require minor hardware modifications in the activation technique. Specifically, the electrical activation will require scaling and use of a small chamber to realize an O₂ concentration change. Electrical activity will be measured for ball-milled material. Given uncertainties associated with electrical contact within a ball-milling canister, transfer of the ball-milled material within a glovebox will be required. Carbothermal (chemical) activation is perhaps the most straightforward to measure by O₂ uptake and conductivity change.

Another area of activity not addressed by the present methods is magnetic activity. Such activity will have impact upon adhesion to metal surfaces, electromagnetic radiation penetration for possible regolith processing, or electromagnetic shielding that may impact communications. While such effects may be rather small, based on the mass percentage of elemental iron, the presence of iron as nanoparticles may impart superparamagnetic properties.

If lunar dust has high electrical charge, then beyond electrical activity, high charge density coupled with mobility of the charge carriers will dramatically increase the dielectric properties of the material. In short, lunar dust may possess characteristics of a conductor rather than an insulator.

In the next phase of this project, efforts would be directed towards combining activation methods. This is a necessity given the concurrence of weathering processes as described in the Introduction. The combined effects of activation methods are unlikely to cancel each other; they may be expected to enhance chemical, electrical, and mechanical activities.

Originally, a quartz crystal monitor was anticipated to characterize the reactive uptake of gaseous O₂ or H₂O by activated material. While possessing high sensitivity, the process of transferring activated material as produced by plasma or carbothermal processing requires further definition. In each of these processes, the material is held upon some surface. Mechanical manipulation could be arranged to deposit activated material to the surface of the crystal monitor. The key issue is that deposition could lead to biasing of particle size or composition depending upon what material may stick to the original surface versus bouncing off the quartz substrate of the thickness monitor in the deposition process. A secondary issue is that quantification of reactive uptake per unit surface area will require an a priori measurement of surface area and the assumption that the chosen activation method does not appreciably change this value. In its favor, reactive uptake per unit mass of simulant is straightforward, providing that the density of the material in powder form is known.

Appendix

In order to provide the reader with an introduction to the apparatuses required for different activation methods, photographs of representative hardware are provided. Figure 1 shows a chamber, gas lines, pressure gauges, and associated electrical instrumentation for characterizing the dust activation. Figure 2 shows a thermogravimetric analyzer. Mass loss upon temperature programmed reduction and mass gain during temperature programmed oxidation can be measured in this instrument. However, electrical characterization of the degree of activation is not possible. Our plans are to perform thermal and chemical activation processing in a furnace with high-temperature electrical leads for conductivity characterization. Still, such temperature-programmed reduction and temperature-programmed oxidation tests can provide a quantitative measure of the reducible component of the simulant. Figure 3 shows a planetary ball mill. The “mixing” bowl containing balls and simulant are inserted inside the apparatus. Rotation rate (and hence impact energy) and milling time are programmable. As indicated in the text, modifications were made to the milling bowl for quantification of oxygen uptake by activated material.

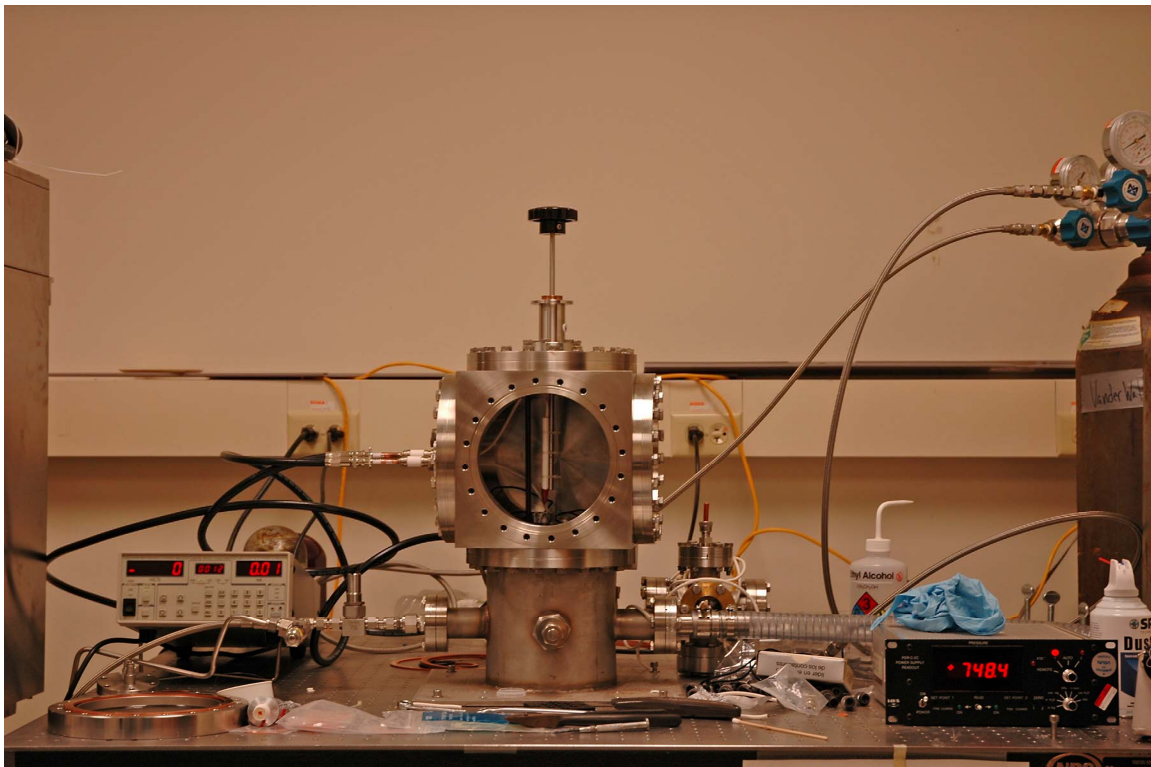


Figure 1.—Chamber for plasma activation. Shown are chamber, electrical feedthroughs, inert gas supplies, plasma power supply, and pressure gauge.

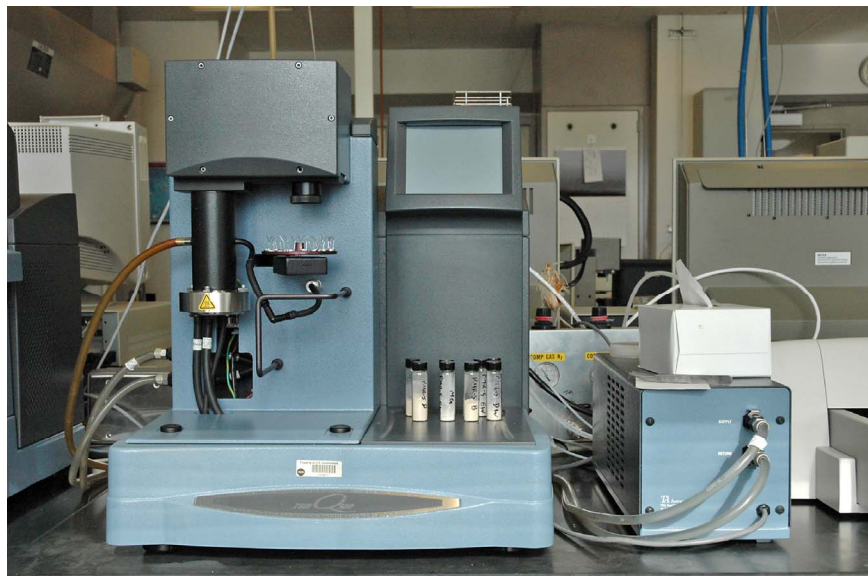


Figure 2.—The thermogravimetric analysis instrument for quantification of the simulant weight loss during carbothermal reduction and subsequent temperature-programmed oxidation.

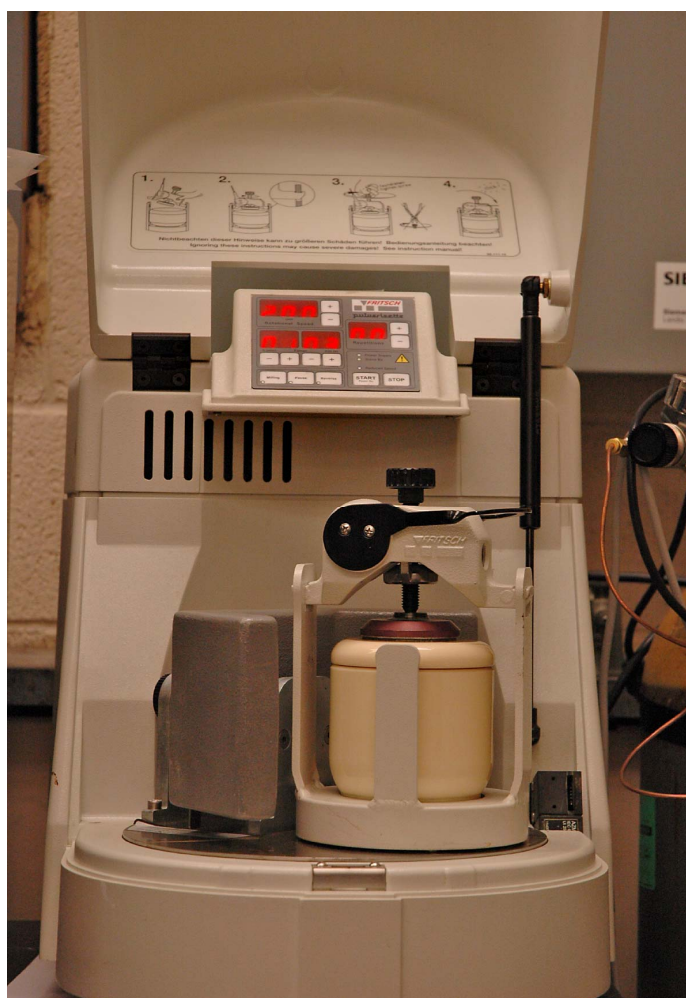


Figure 3.—The Fritsch high-energy ball mill with representative milling bowl.

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