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James R. Gaier
Glenn Research Center, Cleveland, Ohio

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James R. Gaier
Glenn Research Center, Cleveland, Ohio

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National Aeronautics and
Space Administration

Glenn Research Center
Cleveland, Ohio 44135

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James R. Gaier
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Abstract

Activation of the surfaces of lunar regolith particles can occur through interactions with solar electromagnetic radiation, solar and galactic particle radiation and micrometeoroid bombardment. An attempt has been made to quantify the relative importance of each of those effects. The effects of these activated surfaces may be to enhance the adhesion and toxicity of the particles. Also key to the importance of activation is the lifetimes of activated states in various environments which is controlled by their passivation rate as well as their activation rate. Although techniques exist to characterize the extent of activation of particles in biological system, it is important to be able to quantify the activation state on the lunar surface, in ground-test vacuum systems, and in habitat atmospheres as well.

Introduction

The surface of the Moon is covered by a layer of crushed rock from 8 to 48 m deep known as the lunar regolith (Ref. 1). The composition of the regolith samples returned from the six Apollo sites and the two Luna sites have been studied in great detail. These are summarized in the Lunar Sourcebook, (Ref. 2) and numerous other references. From these studies it is known that 65 to 90 percent of the regolith is composed of silicate minerals, depending on location on the lunar surface. The largest component is made of plagioclase feldspars [(Ca-Na)(AlSi)₄O₈], followed by pyroxenes [(Ca-Fe-Mg)Si₁₂O₆] and then olivine [(Mg-Fe)₂SiO₄]. Oxide minerals such as ilmenite [(Mg-Fe)TiO₃] and spinels [FeCr₂O₄, FeTiO₄, FeAl₂O₄, MgTiO₄] are present as well. Although the spinels occur in only trace amounts, they are extremely hard and may be important abrasive agents. Ilmenite, which can act as an oxygen ore, has been found in mare basalts in concentrations as high as 20 percent, but only about 0.5 percent in the highlands. Volcanic and impact glasses can make up from about 7 to 30 percent of the regolith. In addition there are small amounts of native metals [Fe⁰, Ni⁰, Co⁰]; phosphate minerals, primarily whitlockite (Ca₃(PO₄)₂); and sulfur, primarily in the form of troilite [FeS]. Some soils contain small amounts of material high in potassium (generally much less abundant on the Moon than on Earth), rare Earth elements, and phosphorus referred to as KREEP. In addition there are about 100 other minerals present in trace amounts, some of which have not been observed terrestrially.

It is thought that the lunar regolith is formed by a series of highly energetic processes known as space weathering.

Energetic solar particles and galactic cosmic rays constantly bombard the lunar surface. This results in the implantation of atoms, primarily hydrogen and helium, as well as sputtering of atoms off of surface of particles. Meteoroids continually strike the surface as well. Even though the vast majority of these are tiny (<1 mm) they strike the surface with so much kinetic energy that they both break up rock and melt it. A series of complex processes follows from this that includes the release of adsorbed and implanted hydrogen and helium, the release of oxygen from the rock structure, the reduction, vaporization and redeposition of iron, and the flow of molten rock downward to form agglutinates and impact breccias.

Regolith Activation

One of the results of these energetic processes is that a relatively large number of highly reactive surface atoms are thought to be formed. Such surfaces are said to be “activated”. Activation includes any process that enhances the chemical reactivity of the surface. Activated states can result from three basic mechanisms. The first is the ejection of electrons from the surface by light in the ultraviolet or x-ray regions (0.01 to 300 nm wavelengths) resulting in free radicals, that is, atoms with unpaired electrons. These are usually chemically unstable and will react with the first atomic collision after they are formed. The second is the sputtering of atoms off of the surface, which often results in the remaining surface atoms having “dangling bonds”, or unsatisfied valence shell electrons. These are also chemically reactive. Third, high energy collisions with micrometeorites or energetic particles ejected from the Sun can result in crystal lattice defects, with atomic displacements which can lead to highly strained structures. These highly strained structures are also chemically very reactive.

Passivation is the process of relaxation of atoms back to the ground state. These processes are dominated by collisions with low energy atoms, but also include spontaneous emission of light, or radiationless relaxation processes. Because of the ultrahigh vacuum present on the Moon, 10⁻¹² Torr during the day and 10⁻¹⁴ Torr during the night, there are few collisions with gas phase atoms to relax or passivate these states. Thus, it is expected that the concentration of these activated states can become significant.

The presence of activated surface states may materially change some of the properties of regolith particles. This is expected to be most significant for the smallest particles (i.e., dust), since they will have the highest surface to volume ratios. Adhesion of the dust to other materials, including spacecraft

components, is expected to increase as the probability of forming covalent bonds from free radicals and dangling bonds is higher. This would make it more difficult to clean the dust from sensitive spacecraft surfaces. If the activated dust is brought into contact with the humid air of the cabin, it might accelerate corrosion or even might catalyze unwanted reactions of trace gases in the atmosphere. Finally, there is also a possibility that the toxicity of the dust could be dramatically increased, as happens terrestrially with silica (Ref. 3).

The technologically important question is not whether the dust is activated, but rather, if it is activated what are the consequences? For example, it is reasonable to assume that the adhesion forces between the regolith and a sensitive spacecraft surface (such as a radiator or a photovoltaic array) will increase since there will be more opportunity for chemical bond formation when one or both of those surfaces is chemically active, but will it change the performance of the surface or the techniques to clean it?

It is well known that adhesive forces may increase a hundredfold or more in ultrahigh vacuum due to clean body effects (Ref. 4). But it is not yet known whether activation will significantly increase the adhesion even more. But if it does and we understand the mechanisms by which the adhesion is enhanced, it may point the way to mitigation technologies. For example, cleaning off dust in an airlock that has been partially pressurized may be much more effective than cleaning in the hard vacuum of the Moon because a large fraction of the active sites may be passivated before the cleaning process begins.

If the effects of activated dust on spacecraft components and health are to be studied, techniques must be developed to measure the degree of activation, and to generate activated dust. But the detection and quantification of activated surfaces is not a trivial matter. Physical changes, such as the displacement of surface atoms and the imaging of defect sites can be done using atomic force microscopy (AFM), transmission electron microscopy (TEM), field effect transmission electron microscopy (FETEM), or electron diffraction. However, these techniques can also impart surface damage of their own, and it is difficult to unambiguously assign the cause to a particular defect. They are also time consuming and can only be done after the test is completed. What is needed is a diagnostic test that can be carried out *in situ* so that the activation state of the lunar regolith or lunar regolith simulants sample can be verified before it is used to test hardware components.

One test that has been proposed is the utilization of a quartz crystal microbalance (QCM). These instruments are very sensitive to mass changes. So if a surface is activated it will react with a reactive gas, such as oxygen, to a greater extent than a passivated surface would. Such an additional mass uptake may well be measurable using a QCM.

A novel technique to quantify the activation of lunar dust simulants has been explored by Vander Wal et al., who monitored the electrical conductivity of the dust as it was subjected to a discharge plasma in a vacuum chamber (Ref. 5).

Although much work remains to quantify this effect, it is relatively straightforward to implement within a vacuum chamber, and so holds promise as an *in situ* activation detector. One of the major obstacles is that a quantitative measure of the activation state of the dust on the lunar surface itself is unknown. This technique lends itself to an instrument that could be mounted on a lunar lander to determine the dynamic changes in activation state of dust on the lunar surface.

In all likelihood some fraction of dust will be tracked from the lunar surface into the lunar habitat and pressurized rover cabins. The activated surface of this dust can react with oxygen and water vapor to form hydroxyl (OH) peroxy radicals (OOH). These are very reactive and corrosive agents that if present in the cabin atmosphere in high enough concentrations over long enough times can corrode metals and embrittle polymers.

Dust activation is also of intense interest to NASA's toxicology community due to the possibility that activation can significantly enhance the toxicity of lunar dust. In this case, the questions revolve around the passivation mechanisms in the humid air within the lung. If the dust is passivated in air quickly, then whether activation enhances the toxicity is less important than if the passivation times are long enough to affect human exposure. If the species persists long enough to be transported into the lung, then as they passivate they may generate reactive oxygen species such as hydroxyl and peroxy radicals in high enough concentrations to overwhelm the body's defenses and damage biological tissues.

Wallace and co-workers have developed procedures and analytical tests to quantify activation under biological conditions (Ref. 6). The tests rely on the activated surfaces generating reactive hydroxyl radicals, which are then assayed with a fluorescent probe. This is a particularly relevant test as it is these secondary products like hydroxyl and peroxy radicals that do the most damage to biological systems.

Evidence For Regolith Activation

There is experimental evidence that the lunar regolith exists on the lunar surface in an activated state. For example, Hoyt, et al., were able to reconstruct the solar flare proton spectrum over the past 5000 yr by studying the thermoluminescence caused by radiation damage (Ref. 7). They found radiation damage (lattice dislocations) in the top 18 mm of lunar rock, decreasing towards the bottom. Most of the damage was attributable to solar flare protons. Cosmic ray damage was found even deeper within the rocks.

In another example, Grossman, et al., measured the cohesion in a porous friable microbreccia in Apollo 11 sample 10065-33 in a vacuum of 7×10^{-10} Torr (Ref. 8). Upon cleaving the sample the cohesion between the two pieces was measured to be 8×10^{-3} N. After just 4 min the cohesion dropped to 2×10^{-3} N, and within 15 min the forces were too weak to be measurable. This may be due to quenching of activated sites by gas

molecules. Although tested at 10^{-10} Torr, the pressure is still two to four orders of magnitude higher than lunar pressures.

Further evidence of regolith activation comes from the experience of the Apollo astronauts. Several of them remarked that when they first took their helmets off after being on the lunar surface that the lunar dust had a pungent odor, often described as the smell of burnt gunpowder. This smell can be the result of olfactory cells responding to free radicals, such as NO_x (a stable free radical) which has a pungent odor as well. Further, the lunar curator reported at the Biological Effects of Lunar Dust Workshop in 2005 that “the gunpowder smell went away in a few hours (Ref. 9).” This would be consistent with the passivation of free radicals on the particle surfaces.

Lunar Simulants

In order to simulate the effects of the particle surface activation four conditions must be met. First, the particles being activated must have the correct bulk composition and chemistry. Starting with materials that have a different elemental composition, or even with materials with the same composition but a different crystal form will not lead to the same surface chemistry. Second, activation mechanisms must be used that will produce the same types of surface damage or modification. This does not imply that the activation must be done using the same mechanism. An atom that is a free radical or has dangling bonds or is in a strained lattice position will react the same regardless of how that excited state is formed. Third, the passivation mechanisms must be slow enough that the activated dust has time to react with whatever other substances or surfaces. Particles activated in air, for example, are likely to react with the surrounding atmosphere long before reaching the substance of interest, given that there are about 30 million collisions per second for a gas at atmospheric pressure (Fig. 1). Last, there must be some way to detect the presence of activated surface species, so that effects seen can be rightly attributed.

Due to the paucity of lunar material available on Earth, most work will necessarily be carried out using lunar simulants. There are several lunar simulants that are available, and more are being developed. Probably the most used simulant at the time of writing is JSC-1A, which mimics lunar mare regolith, and the most prominent in development in NU-LHT, which mimics lunar highlands regolith.

Lunar simulants are made from terrestrial rock and minerals, and some common components of terrestrial rock are rare or absent in lunar materials. Potassium feldspars $[(\text{K-Al})\text{Si}_3\text{O}_8]$ and silica $[\text{SiO}_2]$ though abundant in terrestrial rock, are rare in the lunar samples. Absent on the Moon (at least in the equatorial regions) because they contain water are micas, clays, and amphiboles. These minerals should likewise be absent from lunar simulants. In addition, the simulant should have a substantial glassy fraction, since much of the lunar regolith is dominated by glasses produced during space weathering. This is important because displacement of surface atoms in a

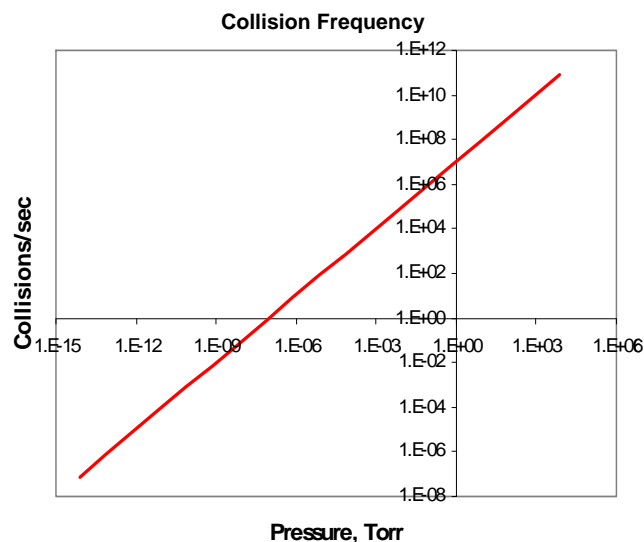


Figure 1.—Collision frequency calculated for N_2 molecules at 298 K as a function of pressure from kinetic molecular theory (Ref. 10). Other molecules in the lunar temperature range give similar values.

highly ordered crystal lattice is a higher energy process than displacement of surface atoms in a glass. For example, if an oxygen atom in a disordered glass structure is bound to a single silicon atom instead of two, calculations show that the energy to remove it from the lattice is decreased by about 5 eV (Ref. 11).

The surface properties of lunar simulant under terrestrial conditions will not mimic the surface properties of lunar regolith under lunar conditions. In fact, lunar regolith will not react the same under terrestrial conditions as it will under lunar conditions. The environment of the Earth is humid, oxidizing and protected from high energy radiation by the Earth’s magnetic field and atmosphere. In contrast, the lunar environment is dry, chemically reducing, and constantly bombarded by high energy electromagnetic and particle radiation. The surface chemistry of any material will be different in these two environments.

The first step in any process to produce activated lunar simulant would be to remove the adsorbed layer of Earth atmospheric gases from the surface of the particles. Such studies are being carried out in the Lunar Dust Adhesion Bell Jar (LDAB). This is a facility built to determine the effects of the lunar environment on adhesion to sensitive spacecraft surfaces. Details of its philosophy and design have been reported elsewhere (Ref. 12). By far the largest contaminant is water vapor, which is almost totally absent from the lunar environment. Studies suggest that heating the simulant *in vacuo* or even in an atmosphere of dry nitrogen to a temperature of 200 °C can accomplish this (Ref. 13). There is an additional problem with water in that most terrestrial rock have inclusions that contain water between the crystalline grains deep within the rock. This may take very high temperatures to remove, but removing it may not be necessary if diffusion of this reservoir of water to the surface is sufficiently slow.

The second largest contaminant is likely organic molecules such as methane, ethane, and hydrocarbon oils that are nearly ubiquitous in the Earth's environment. It is easiest to remove these organics through oxidation. There are many routes to oxidation, but using a gas phase method that will not distort the surface composition of the rock is probably best. Either oxygen plasma (as is used in the LDAB) or ozone generation can result in reactive oxygen that can convert hydrocarbons to volatile carbon dioxide. Since the dominant minerals are aluminosilicates, silicates, and oxides, the surface composition will likely be unchanged.

The oxidation processes described above will leave the surface highly oxidized. This means that transition metals such as iron will likely be in very high oxidation states, such as iron (III). The lunar surface, on the other hand, is being bombarded by a constant stream of hydrogen and helium atoms and ions from the solar wind, which generate a highly reduced surface. Iron (III) is almost unknown on the lunar surface, with the iron being predominantly in the iron (II) state, or even in the iron (0) state. So this preponderance of water and oxygen that is found adsorbed to the surface of terrestrial materials must be replaced with a preponderance of hydrogen.

After a correct bulk material has been obtained, the terrestrial surface layer stripped off, and lunar-like surface layer has been added, then the simulatant can be activated. There are many ways this can be accomplished. In the LDAB the dust is subjected to a second plasma, where the gas is a 6 percent hydrogen in helium mixture. Although the solar wind is about 96 percent protons and 4 percent α particles, the much higher helium content was selected for safety reasons. It has also been found that the sticking coefficient of hydrogen is so much higher than helium that hydrogen dominates the surface adsorption despite its lower concentration. Whenever the dust is stirred, sifted, or crushed in the LDAB, hydrogen is released but helium is not.

Mechanisms of Activation

As illustrated in Table I, typical mineral bonds have strengths that range from 3 to 9 eV (Ref. 14). Thus any mechanism that can impart more than about 3 eV to a surface atom has the potential to activate it.

TABLE I.—ENERGY OF OXIDES FOUND IN LUNAR REGOLITH[†]

eV	ΔH_{bond}	ΔH_{form}	$\Delta H_{\text{lattice}}$	$\Delta H_{\text{sublimation}}$	$\Delta H_{\text{ionization}}$
SiO ₂	8.15	-9.44	136	4.66	45.1
TiO ₂	7.28	-9.79	126	4.90	43.3
Al ₂ O ₃	4.98	-17.4	165	3.42	28.5
CaO	4.77	-6.58	35.4	1.84	11.9
Cr ₂ O ₃	4.37	-11.8	158	4.11	31.0
MgO	3.90	-8.20	39.3	1.52	15.0
Na ₂ O	3.34	-4.29	25.7	1.11	5.14
FeO	1.60	-2.82	39.3	4.32	16.2

[†]Although the metals are largely bound to oxygen in the mineral crystal lattices, and traditionally expressed as oxides as in this table, it is not correct to think of the minerals as being a simple collection of metal oxides, as their structure is much more complex.

One potential energy source is the light energy emitted by the Sun. The Sun is a much more potent source of ultraviolet radiation on the Moon than the Earth because a substantial portion of the solar electromagnetic output is filtered out by the atmosphere of Earth, much of it by the ozone layer. A plot of the standard "air mass zero" is shown in Figure 2 (Ref. 15). This is the spectrum observed just above the Earth's atmosphere, and is what is seen on the lunar surface as well. Note that the bulk of the radiation is in the visible and infrared regions of the spectrum, between about 0.2 and 7 eV.

Work by Sternovsky et al., found that the work function (energy to extract an electron from the surface) of the lunar regolith can be expected to be about 5.8 eV (Ref. 16). Analysis of the spectrum of the solar energy reaching the lunar surface reveals that about 0.1 percent of the energy released by the Sun is in the form of photons that have energy greater than 5.8 eV (wavelength <213 nm), enough energy to remove an electron and form a free radical. Given that the intensity of the solar flux is 1366 W/m², on average each surface atom will be hit by a photon with more than 5.8 eV of energy about once every 2 min.

In the LDAB there are two processes that generate UV light with more than 5.8 eV. The first is through two radio frequency plasmas used to prepare the dust. The first plasma is an air plasma used to oxidize away organic matter contaminating the dust. The second plasma is a hydrogen-helium plasma used to chemically reduce the surface states. Analysis of the emission spectrum reveals that about 1 percent of the light given off has an energy greater than 5.8 eV. Each plasma has an input power of 200 W applied for 1 hr, or a total of 720 kJ. However, it is not known how the energy is partitioned between ionization and excitation of electronic states, or what fraction of the light impinges on the dust, so the flux cannot be calculated.

The second UV generating process is through heating the dust coated samples with a Xe arc lamp. The intensity of the LDAB arc lamp is about 20 Suns, so following from the

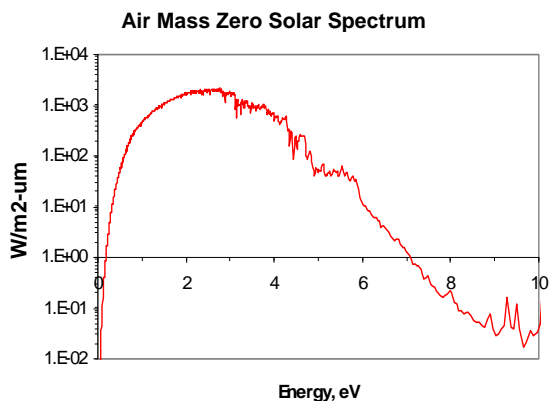


Figure 2.—Air mass zero solar spectrum over its most intense energy region (Ref. 13).

estimation above, each atom should be exposed to a phonon with 5.7 eV energy or higher about every 6 sec. It should be noted that the dust is only exposed to that flux for about 15 min during the test.

In addition to electromagnetic radiation, the Sun also bombards the lunar surface with high energy particles, largely protons, but α particles as well. By far the greatest number of these come from the solar wind, but solar storms and solar flares bombard the surface with less frequent, but higher energy particles. In addition, the lunar surface is also bathed in a low flux of very high energy cosmic rays.

Figure 3, adapted from Wilson, et al., shows the particle radiation flux density and energy emitted by the Sun to the Moon (Ref. 17). A typical mineral crystal will have about 10^{15} atoms/cm² of surface. Extrapolation of the solar wind protons to that value leads to the conclusion that a solar wind proton will strike a surface atom carrying a keV or more of energy each (24 hr) day. Similarly, it will interact with a 1 MeV solar storm proton about once per millennia, a 100 MeV proton, and a GeV cosmic ray about once every 100 million years. These energetic particles may have several collisions within the lattice before they come to rest, and will often become implanted, as evidenced by their release as hydrogen gas from the Apollo samples (Ref. 18). They also sputter atoms from the surfaces of the particle, resulting in dangling bonds and lattice dislocations.

High energy particle radiation is simulated in the LDAB by the radio frequency plasmas described previously. Again, it is difficult to know how the total energy is partitioned between electromagnetic radiation and the ion formation and accelerations. Further, it is not known what fraction of the ions strike the dust, nor with what energy. An in situ instrument that could quantify the extent of activation would be useful in determining how long the dust should be exposed to the plasma.

The meteoroid flux that the International Space Station is exposed to has been quantified by Christiansen (Ref. 19). A log-linear extrapolation of his Figure 1-2 to smaller particles sizes shown in Figure 4 has been used to estimate the micrometeoroid flux on the lunar surface.

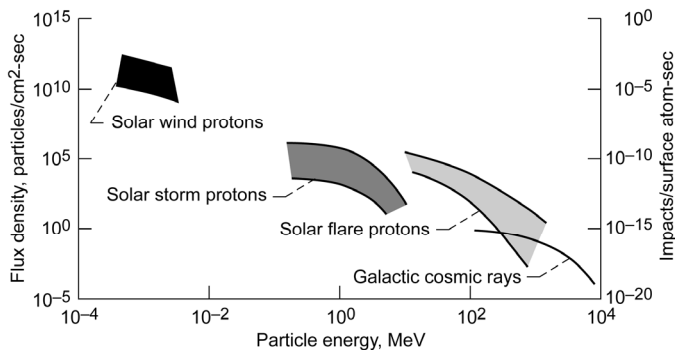


Figure 3.—Impacts per lunar surface atom per second for energetic radiation. (Adapted from Wilson, et al. (Ref. 3).)

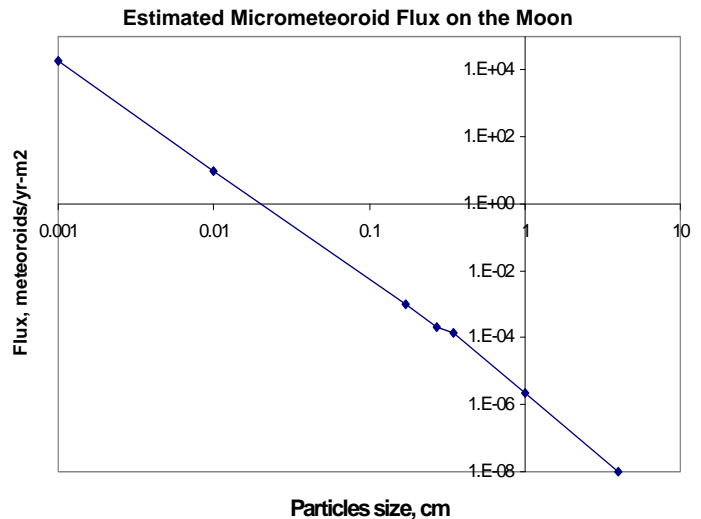


Figure 4.—Impacts on lunar surface per year per m². (Adapted from Christiansen (Ref. 16).)

It is assumed that the flux in low Earth orbit is similar to that of the lunar surface. It is recognized that because of the greater size of Earth's gravity well, the flux on the lunar surface is probably somewhat lower. The estimate is that each year there will be about ten 100- μ m meteoroids and about ten thousand 10- μ m meteoroids strike each square meter of surface.

Each of these high energy collisions with the regolith will result in the fracture of a large number of particles. The surfaces of freshly ground minerals are characterized by the presence of highly reactive free radicals (Ref. 3), and since the regolith was formed by impact fracture, they probably form on the lunar surface as well. These fracture surfaces often include "shock structures" which include dislocations and dangling bonds which will be chemically active. Thermal excursions which can be as large as 300 K near the equator will induce thermal stresses which may create new dislocations and fracture surfaces.

Micrometeor impacts can be simulated by fracturing the simulant using high impact milling. As the grains fracture, defect sites will be present that are induced by the strain before fracture. Although this method may be used to activate dust or test of reactivity in the habitat or toxicological environment, it would be difficult to generate this type of activation in a vacuum environment. That being said, Grossman et al.'s study referred to above induced a single fracture of a relatively large rock to study the nature of fracture activation (Ref. 7).

Mechanisms of Passivation

While there are many processes on the lunar surface to activate particle surfaces, there are not many passivation mechanisms. Under the rarified atmosphere (actually a ballistic exosphere) with pressures of 10^{-12} Torr on the day side and 10^{-14} Torr on the night side, there are very few collisions with gaseous atoms that have a chance to relax the active site. Such atoms as do collide will have such low collisional energy that

few will be able to heal a lattice defect, as is illustrated in Hoyt's study referred to above (Ref. 6). As a result, passivation in the lunar environment is expected to be a very slow process.

The work to produce the highly activated surfaces in ground test systems will be for naught if passivation occurs before the active surface can react with the experimental surface in question. One possibility is auto-passivation, that is energy can be emitted to return an excited atom to the ground state, or a lattice defect may spontaneously correct itself. But more often passivation will be the result of collisions with other atoms. So the most effective way to limit passivation is to limit the exposure of the activated material to external atomic collisions. Figure 1 shows a plot of molecular collisions/sec as a function of pressure. Examination of the collision frequency reveals that effectively, this means by operating in a vacuum.

Each type of activation site will have its own passivation energy. Free radicals have such a low passivation energy that they are usually passivated by the first collision, whereas that of lattice defects is so high that they can be used to track solar activity over thousands of years (Ref. 3). The probability that a collision will be by an atom or molecule with sufficient energy is provided by the Boltzmann distribution, which is very sensitive to temperature. So the best condition to extend the lifetime of the activated state is low pressure and low temperature.

The LDAB tests are carried out in a vacuum of 10^{-8} Torr. At these pressures the passivation of the particle surfaces will be slowed, but the pressure is still 10,000 times higher than even the dayside pressure on the Moon. Tests must be carried out in UHV facilities at 10^{-12} Torr to determine how these passivation rates compare with those at lunar pressures.

Conclusions

The lunar surface is an energetic environment. Electromagnetic and particle radiations emanating from the Sun contain a small fraction of photons and protons that are energetic enough to create free radicals, dangling bonds, and lattice defects. There is circumstantial evidence from the Apollo record that these activation processes do occur. These high energy activation sites can affect the reactivity of the dust particle surface enough to affect their adhesion to spacecraft surfaces and human toxicology.

Avenues are being explored to generate activated dust surfaces using mechanical, thermal, and chemical means. Progress is being made on the quantification of the activation state under high vacuum conditions. Methods to quantify their potential for toxicological effects are already well known.

It is important to quantify the passivation rates in a variety of environments. Since they are dependent on molecular collisions they will vary widely depending on whether their environment of interest is the lunar surface, standard ground-test vacuum systems, airlocks, habitats, or within the lung. Overall it is important to understand all aspects of activated states on dust

particles; how they are formed, how the effect mechanical and biological systems, and how they are passivated.

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