Nanophase Fe⁰ in lunar soils

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Back scattered electron and transmission electron imaging of lunar soil grains reveal an abundance of submicrometer-sized pure Fe⁰ globules that occur in the rinds of many soil grains and in the submillimeter sized vesicular glass-cemented grains called agglutinates. Grain rinds are amorphous silicates that were deposited on grains exposed at the lunar surface from transient vapors produced by hypervelocity micrometeorite impacts. Fe⁰ may have dissociated from Fe-compounds in a high temperature (>3000°C) vapor phase and then condensed as globules on grain surfaces. The agglutinitic glass is a quenched product of silicate melts, also produced by micrometeorite impacts on lunar soils. Reduction by solar wind hydrogen in agglutinitic melts may have produced immiscible droplets that solidified as globules. The exact mechanism of formation of such Fe⁰ globules in lunar soils remains unresolved.

1. Introduction

More than 30 years of sample research, many more years of remote investigation, and recent flyby missions have characterized the moon's basic geological properties. The radius of the moon is about 1740 km. Arguably, it has a 35–40 km or 60–70 km thick crust above an approximately 1200 km thick differentiated mantle (Khan *et al* 2000; Khan and Mosegaard 2001). The mantle, however is solid, has no asthenosphere and there is no evidence of any plate motion ever. The last volcanic eruption, dated by crater counting and remote sensing methods, may have taken place as late as only 1 b.y. ago (Spudis 1996). The moon may have a core of primitive material or a solid Fe–Ni core that may be as much 500 km thick (Taylor 1982). The moon has neither an atmosphere nor a hydrosphere. Meteorites of all sizes, large and small, have freely bombarded the lunar crust at hypervelocity; micrometeorites continue to pound and pulverize its surface and modify its regolith. The average grain size of the surficial material of the moon is about 50 µm. The dust produced by the impacts is extremely fine and the grain-size distribution of the submillimeter fraction of the lunar regolith is

skewed towards the finest end. Note that the dust carried into the Apollo 12 lander cabin on the boots and spacesuits of astronauts interfered with visibility and breathing after liftoff (Bean *et al* 1970). In the absence of a lunar magnetosphere, solar wind ions also bombard its surface freely and implant a high dose of protons in the rinds of exposed grains. For a general overview, see Taylor (1982); Heiken *et al* (1991); and Spudis (1996).

The lunar regolith is principally produced by meteoritic bombardment of the lunar surface and its properties are products of several complex processes (McKay *et al* 1991). Large impacts excavate fresh material from below and replenish the regolith, commonly burying pre-existing regolith with the new ejecta. Smaller impacts redistribute the existing regolith churning them over. Micrometeorites bombard all available targets on the moon; those that bombard the submillimeter fraction of the regolith, commonly known as lunar soil, effectively mix soil components locally. All hypervelocity impacts vaporize a part of both the projectile as well as target. A part of this vapor escapes from the moon while elements or compounds with lower condensation temperatures condense back on the surfaces of exposed grains as a thin film. As

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mentioned above, solar wind ions are implanted and concentrated in the outer rinds of the soil grains; these rinds may be as thick as 20 nm. Thus, the outer rinds of grains exposed at the lunar surface acquire different properties. The longer the exposure of the grain the greater, for example, is the concentration of solar wind ions in its rind. The degree of change in lunar grains, or a soil as a whole, in response primarily to exposure at the surface is called its maturity (McKay *et al* 1974; Arnold 1975).

As a lunar soil matures some of the soil grains, impacted by a micrometeorite, melt. The impact melt is turbulent and somewhat frothy as solar wind implanted gases boil and escape (or are stored in vesicles); the melt has a very small volume, possibly in the order of 0.1 mm^3 . Each melt quenches quickly but not before collecting and incorporating other grains in the melt. Some of the finest dusty grains are entrained in the flow within the melt. Imaging at high magnification, such as our current work, has shown that submicrometer-sized pure Fe^0 globules occur along with dust grains and mark flow lines. Previous optical microscopic observation, limited at $\sim 1 \mu\text{m}$ resolution, had not realized the abundance of such small Fe^0 globules. These micrometeorite-impact-melt-cemented composite and constructional vesicular grains, commonly smaller than a millimeter, are called agglutinates (figures 1 and 2). The melt and the glass are called agglutinitic melt and agglutinitic glass respectively. Naturally, the agglutinate content of soils increases with increasing maturity (McKay *et al* 1974; Basu 1977; McKay and Basu 1983).

2. Unresolved questions about the origin of Fe^0

The origin of the nanoscale pure Fe^0 globules is not quite known. Two competing hypotheses regarding the mechanism of formation of Fe^0 are currently being debated. This paper discusses how our current work bears on the problem. One hypothesis, prevalent for a long time, envisages reduction of Fe-bearing phases (e.g., ilmenite) in the agglutinitic melt in the presence of solar wind hydrogen that had been implanted in soil grains (Housley *et al* 1972, 1974; Morris 1977, 1980; Taylor and Cirlin 1985). The reaction envisaged involving an oxide component would be in the form

or

The other, revived more recently, envisages dissociation of Fe from Fe-bearing phases in a high-

Figure 1. Back scattered electron (BSE) image (76321-262) of a polished thin section of an agglutinate showing the general morphology of agglutinates, distribution of mineral and rock clasts marked as Px (pyroxene), Pl (plagioclase), Il (ilmenite), and BT (basalt); vesicles (V) are nearly circular; clusters of nanoscale Fe^0 , unresolvable at this scale, and a few larger Fe^0 globules are marked with arrows; scale bar is $10 \mu\text{m}$ long.

Figure 2. BSE image (77531-1) of a polished thin section of an agglutinate showing the distribution Fe^0 globules (bright) defining flow lines (marked with arrows; the box is the location of figure 5); parts of the glass are very rich in Fe^0 globules (upper left half); notice that large vesicles may render some larger agglutinates rather fragile; scale bar is $10 \mu\text{m}$ long.

temperature (e.g., $>3000^\circ\text{C}$) vapor produced by impacts followed by condensation of Fe^0 globules on the surfaces of exposed grains in lunar soils (Hapke 1975, 2001; Keller and McKay 1993, 1997; Christofferson *et al* 1996; Yaklovlev *et al* 2002). The reaction would be simpler such as

Note, however, that metals other than Fe should also dissociate in such high temperature vapor (Bhandari, personal communication). Apparently pure-K or extremely K-rich spherules ($\sim 1\text{--}5\text{ }\mu\text{m}$ in diameter) observed in Luna 20 soils (Bhandari and Shah 1979) could be products of such dissociation. We have not observed any metal other than Fe⁰ in the agglutinates we have studied. Nor is there any other report of pure metal globules in agglutinitic glass or in amorphous rinds of lunar grains. Such absence does not negate the hypothesis because high-magnification observation for reduced metal in agglutinates has just started and it is possible that the less common, if any, would be identified soon. Regardless, both processes should release a vapor phase intermittently. There is no monitor on the moon to await intermittent micrometeoritic impact on lunar soils and detect if miniscule amounts (OH), H₂O or O₂ is released, which are susceptible to immediate ionization in the lunar environment. Therefore, only indirect methods can test the competitive advantage of one hypothesis over the other.

3. Data and models: Current status

There is agreement on some qualitative observations. Many Fe⁰ globules, especially those that are $>1\text{ }\mu\text{m}$ in size, occur in agglutinitic glass. Many Fe⁰ globules, especially those in the $<25\text{ nm}$ size range, are seen to reside in an amorphous coating on lunar grains. Flow lines are ubiquitous in agglutinitic glass, indicating a liquid (melt) precursor. The amorphous coatings on soil grains are very thin and do not show flow lines, indicating that some vapor phase might have been the precursor of the coatings.

3.1 Imaging

Secondary electron (SE) images from scanning electron microscopes (SEMs) revealed many morphological features of grain surfaces of the lunar regolith and spawned the concept of weathering in the lunar environment (e.g., Cloud *et al* 1970; Margolis *et al* 1971). SE imaging helped to discover vapor deposited euhedral Fe⁰ crystals in vugs of lunar breccias (Clanton *et al* 1973), trains of Fe⁰ beads splashed (?) on surfaces of agglutinates (McKay *et al* 1991; their fig. 7.2f on p. 297) as well as those embedded in the agglutinitic glass. Transmission electron microscope (TEM) images of extremely thinly sliced lunar grains (ultramicrotomed to about 70 nm) show that in addition to the mode of occurrence of Fe⁰ globules mentioned above, they also occur in the outer amorphous rinds of the grains (Keller and McKay 1992;

their fig. 5 on p. 139; Christofferson *et al* 1996). Microtoming and TEM imaging are extremely time-consuming. Hence, routine imaging and measurements of a large number of Fe⁰ globules are not possible yet. However, some painstaking work has indicated that the mean grain size (number frequency) of the sub-30 nm population of Fe⁰ globules in agglutinitic glass is about 7 nm and that in the amorphous rinds is about 3 nm (Keller and Clemmet 2001).

Backscattered electron (BSE) imaging of polished grain mounts of lunar soils is relatively less time consuming than TEM imaging. Current generation of SEMs can now produce BSE images at nanometer-scale resolution ($\sim 4\text{ nm}$) given enough contrast in average atomic numbers of adjacent phases. We have used a field emission gun SEM to obtain BSE images of agglutinitic glass in several lunar soils and have recorded the distribution of Fe⁰ globules (figures 3 and 4). Data obtained so far on some 13500+ globules in agglutinates in 12 soils show that pure Fe⁰ globules are all $<1\text{ }\mu\text{m}$ in size, with a mean size of $\sim 90\text{ nm} \pm 7\text{ nm}$ (James *et al* 2001, 2002). The mean size is about the same and the standard deviation of the distribution is also about the same in all of the 12 soils (NASA numbers 15221; 15601; 61161; 61181; 61221; 63501; 64501; 67941; 72151; 75081; 78421; 79221). These soils represent the spectrum of lunar soils compositions developed on pure mare basalt to nearly

Figure 3. BSE image (74121-139) of a polished thin section of an agglutinate especially emphasizing trains of very small Fe⁰ globules in relatively Fe-rich glass in which clasts of pyroxene (light gray, right of center) and plagioclase (dark, fractured, bottom center) are incorporated; most of the small globules ($<100\text{ nm}$) are not resolvable at this scale (but see figure 6); scale bar is $10\text{ }\mu\text{m}$ long.

Figure 4. BSE image (61181-43) of a polished thin section of an agglutinate emphasizing the way trains of very small Fe^0 globules wrap around a clast; the clast hosting larger Fe^0 globules is feldspathic glass (FG); scale bar is $1\ \mu\text{m}$ long and none of the globules are $>1\ \mu\text{m}$ in size.

pure anorthosite; actual $\text{FeO}\%$ in these soils range from 4.2% to 19.2%. The maturity of these soils range from being immature ($I_s/\text{FeO} = 9.2$) to mature ($I_s/\text{FeO} = 92$). The difference in the results between TEM and BSE-SEM data is due to some of the constraints of the techniques. TEM measurements were done on a smaller population of smaller grains ($<30\ \text{nm}$) and hence could not detect larger globules; and, BSE-SEM measurements could not resolve Fe^0 globules $<5\ \text{nm}$ in size.

3.2 Experimental

Many experiments have been conducted to understand impact processes and their products. Only in a few of these experiments porous targets are used, such as sand, to simulate impacts on the lunar regolith. Laboratory experiments show that apart from pulverizing the target, impacts also melt and vaporize the part of the target that suffers peak pressures (Schaal and Hörz 1977, 1980; Stöffler 1975; Schultz 1996; Sugita and Schultz 1999; Crawford and Schultz 1999). Laboratory experiments are carried out under earth's atmospheric conditions; but the lunar environment is under virtual vacuum. Speeds of meteorites impacting the moon or earth range mostly in the region of 15–20 km/s. In laboratories, high velocity vertical guns can barely achieve speeds of up to about 10 km/s. Peak pressures necessary to vaporize silicate targets, such as 100 GPa or higher, have not been achieved in laboratory experiments. Results of experiments on earth have to be scaled up for realistic predictions under lunar

conditions. Therefore, all predictions are *per force* model dependent.

Cintala (1992) has developed a model that enjoys current consensus on thermal effects on the lunar regolith in response to hypervelocity impacts. Results show that hypervelocity impacts (say at $\sim 15\text{--}20\ \text{km/s}$) on the lunar regolith would melt and vaporize the target in a ratio of about 7:1. Some of the vapor escapes into space, some may be encapsulated in the melt as bubbles, and some condense back on the surface of exposed soil grains. As noted above, if the vapor is at a very high temperature (say $>3000^\circ\text{C}$; cf. Yaklovlev *et al* 2002), it would dissociate and Fe^0 globules would condense along with and in an amorphous silicate host.

The amount of melts produced by impact events is far more abundant than vapor. Depending on the time available to quench, crystallites may form. If reducing gases are available, such as solar wind implanted hydrogen, the melt would be reduced and Fe^0 would form, a common phenomenon in metallurgical processes. The reduced pure Fe^0 melt would be immiscible in the silicate melt and surface tension effects would render them as droplets.

Marginally relevant to the issue are a series of experiments in which Fe^0 was produced by forcing a stream of hot hydrogen gas ($\sim 1100^\circ\text{C}$) over a bed of ilmenite or lunar soils (Allen *et al* 1994, 1996). These experiments show that neither dissociation in a high temperature vapor nor reduction in a melt is absolutely necessary to produce Fe^0 . The lunar environment is essentially in a state of hard vacuum with $\sim 10^4$ molecules/ cm^3 in its 'atmosphere' during the day and fewer molecules at night; the earth's atmosphere by contrast has $\sim 2.5 \times 10^{19}$ molecules/ cm^3 (STP) in its atmosphere (Heiken *et al* 1991). Therefore, the lunar environment does not lend itself to a flow of a large quantity of hot hydrogen gas; hence the experiments described above are useful in understanding the general phenomenon of reducing FeO components to Fe^0 , but do not directly contribute to the issue at hand.

4. Discussion

Theoretical deductions, experimental results, empirical observation, and modeling calculations indicate that nanoscale Fe^0 globules can be produced at the lunar surface (1) as condensates from an impact-vapor that has suffered high temperature dissociation, and (2) as immiscible droplets in an impact melt of silicates, which (*i.e.*, the melt) has suffered reduction through solar wind implanted hydrogen in the target. Initially the former occurs exclusively on grain coatings and the

latter in agglutinitic glass. Currently available data on the size distribution of Fe⁰ globules in grain coatings and in agglutinitic glass are fully compatible with the scenario described above. The mean size of Fe⁰ in grain coatings is about 3 nm and the largest is about 12 nm (Keller and Clemmet 2001; TEM measurements), which are compatible with a condensation-from-vapor origin. The same technique also shows that Fe⁰ globules in agglutinates are larger (mean = 7 nm; max = 30 nm). Our BSE-SEM data on Fe⁰ in agglutinitic glass are much more comprehensive and indicate that the maximum size of pure Fe⁰ globules is limited to about 1 μ m and that their mean size is about 105 nm (James *et al* 2001, 2002).

The different size distributions and especially the upper observed limit of $\sim 1 \mu$ m are not easy to understand and interpret. Data show that the larger globules occur exclusively within agglutinitic glass. One possibility is for the small Fe⁰ globules to coagulate and grow bigger after formation and while in the agglutinitic melt. Continued micrometeoritic impacts rework the uppermost part of the lunar regolith (McKay *et al* 1974; Arnold 1975). Reworking includes recycling older agglutinates as well (Basu 1977; McKay and Basu 1983). As older soil grains, including former agglutinates, are melted fully or in part, and are incorporated into newer agglutinates, some pre-existing Fe⁰ globules may be mobilized to coagulate, increase in size, and form larger immiscible droplets. Agglutinitic melts, being very small in volume, quench very rapidly and form silicate glass. Because not much time is available, coagulated immiscible Fe⁰ droplets, regardless of ultimate origin, can only reach a time-limited size (figures 5 and 6).

It will be interesting if one could calculate the rate at which a silicate melt must cool to quench to glass without forming crystallites and yet allow 3 nm Fe⁰ globules to coagulate to 7 nm or larger sizes. Repeated melting, *i.e.*, through repeated recycling of agglutinates (e.g., Basu and Meinschein 1976) would clearly increase the mean size of Fe⁰ globules in agglutinitic glass. As of now we do not have the wherewithal to estimate the proportion of vapor-condensed and immiscible melt derived Fe⁰ globules in agglutinates, especially because most of the former are likely to have been remobilized in the agglutination process. Nanotechnology is not far advanced yet to characterize such small particles and distinguish one from the other. If the kinetics of partitioning of isotopes of iron are different in these two processes, it might be possible to identify the two different products on the basis of $\varepsilon^{57}\text{Fe}$.

In summary, this is an exciting time for investigating the origin of nanophase Fe⁰ and their growth in lunar agglutinates. The late Professor Sukomol

Figure 5. A high magnification view (77531-13) of the box in upper left center of figure 2; notice small globules embedded in Fe-rich glass schlieren (bright, sinuous); scale bar is 1 μ m long.

Figure 6. A very high magnification view (77531-16) of the schlieren in figure 5 showing discrete globules within the schlieren; scale bar is 100 nm long.

Kumar Chanda might have compared this time with that in the early 1960s when investigations into recrystallization of micrite or neomorphism in limestones were just as raw (Chanda 1963).

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