

# Microwave Sintering of Lunar Soil: Properties, Theory, and Practice

Lawrence A. Taylor<sup>1</sup> and Thomas T. Meek<sup>2</sup>

**Abstract:** The unique properties of lunar regolith make for the extreme coupling of the soil to microwave radiation. Space weathering of lunar regolith has produced myriads of nanophase-sized Fe<sup>0</sup> grains set within silicate glass, especially on the surfaces of grains, but also within the abundant agglutinitic glass of the soil. It is possible to melt lunar soil (i.e., 1,200–1,500 °C) in minutes in a normal kitchen-type 2.45 GHz microwave, almost as fast as your tea-water is heated. No lunar simulants exist to study these microwave effects; in fact, previous studies of the effects of microwave radiation on lunar simulants, MLS-1 and JSC-1, have been misleading. Using real Apollo 17 soil has demonstrated the uniqueness of the interaction of microwave radiation with the soil. The applications that can be made of the microwave treatment of lunar soil for in situ resource utilization on the Moon are unlimited.

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## Introduction

The chemical and physical characteristics of lunar soil, the fine fraction (<1 cm) of the lunar regolith, are at the root of the unique properties of this abundant resource on the Moon. Below, these characteristics of the lunar soil are addressed as they make the soil an effective feedstock for microwave processing. Considerations of the science of the properties of fine lunar soil, coupled with the physics of microwave radiation, form the basis for our creating processes for the microwave sintering, hot-pressing, and melting of this soil. Combining both conventional and microwave into hybrid heating can be used to form various structural and mechanical materials for use both on the Moon and in microgravity space. This will allow the effective manufacture of everything from roads on the Moon to parabolic antenna dishes for transport to near lunar orbit (e.g., L1), indeed, materials for construction in space (Taylor and Meek 2004).

## Science of Lunar Soil Formation and Characteristics

Lunar soil is generally fine-grained with over 95 wt % less than 1 mm; 50% is less than about 50 μm (the thickness of a human hair); and 10–20% is finer than 20 μm. The lunar-soil particle-size distribution is very broad: “well-graded” in geotechnical en-

gineering terms, or very poorly sorted in geological terms (Carrier 2003). In addition, because of the irregular, reentrant particle shapes, the specific surface area is high: approximately 0.5 m<sup>2</sup>/g. In fact, lunar soil particles have about eight times more surface area than an assemblage of spheres with the equivalent particle size distribution. As a result of both of these factors, lunar soil particles do not pack together as efficiently as, for example, uniform spheres. Even when lunar soil is packed extremely tightly (by a combination of compression and shaking), the porosity is roughly 40–50%—high by terrestrial standards (Carrier 1991).

## Formation of Lunar Soil

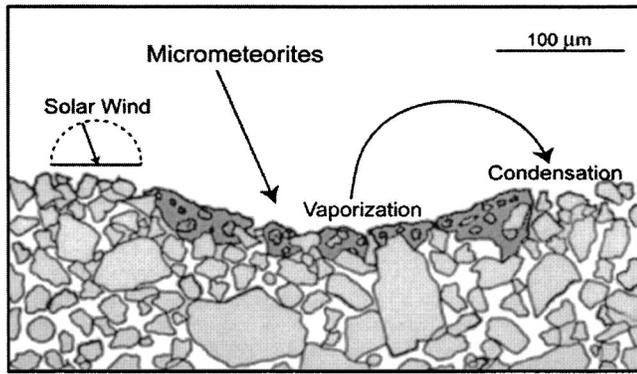
The major factor in the formation of lunar soil is micro-meteorite impact. Rocks are broken into smaller pieces, accompanied by formation of considerable glass (typically 40–75%) from impact-produced melting (Taylor et al. 2001a,b). Two competing processes are operative: *comminution* of larger soil particles into smaller ones, and *agglutination*, where silicate melt welds together soil grains into glassy aggregates (Hu and Taylor 1977; Taylor and McKay 1992). These two competing processes complicate the formational characteristics of the soil, as shown in Fig. 1. The lunar soil consists basically of the disaggregated pieces of the rock (i.e., minerals), but with a large component of impact-produced glass. Although there is some native Fe (elemental Fe<sup>0</sup>) in lunar rocks, the amount of native Fe<sup>0</sup> in the soils is at least 10× greater than in the rocks from which the soil was formed (Taylor and Cirlin 1985; Taylor 1988). At first, it was assumed that this was meteoritic metal. However, it was demonstrated that the amount of total meteoritic contamination to the soil is only about 2% at best, and only a fraction of that consists of metallic iron. It was then reasoned that the majority of the native Fe<sup>0</sup> in the lunar soil was formed by the auto-reduction of the FeO in the silicate melts formed by micro-meteorite impacts (e.g., Housley et al. 1973). Solar-wind implanted protons (hydrogen nuclei) on the surfaces of lunar soil particles reduce FeO in the melt elemental Fe<sup>0</sup> that homogeneously nucleates into myriads of nanophase-sized (3–33 nm) Fe<sup>0</sup> (abbreviated: np-Fe<sup>0</sup>).

This impact-generated melt quenches, thereby forming the

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**Fig. 1.** Schematic diagram illustrating the processes of cominution, agglutination (dark grains), and vaporization in the soil-forming processes on airless bodies (Taylor 1988)

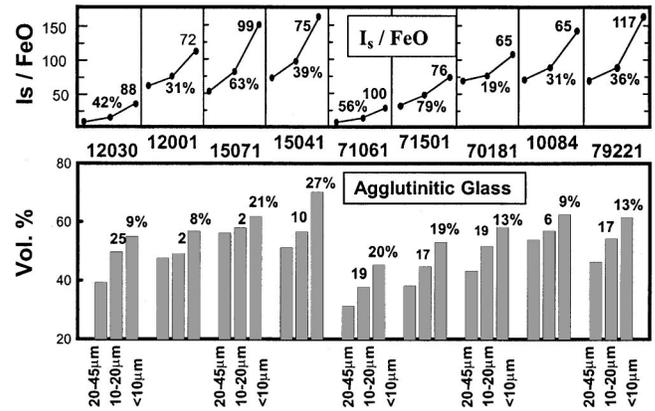
glass that binds together the aggregates of soil particles called “agglutinates.” Most of this fine-grained np-Fe<sup>0</sup> in the agglutinitic glass is not visible with even the best optical microscope, but it gives a strong magnetic susceptibility signature to the fine fraction of lunar soil (Taylor et al. 2001b).

Ferromagnetic resonance (FMR) is used to measure the signal from the np-Fe<sup>0</sup> (Morris 1976, 1978). This measure, designated  $I_s$ , is further divided by the total FeO content of the sample in order to normalize the differences in total iron contents of different samples. The value  $I_s/FeO$  designates the amount of iron in a sample that is in the native Fe<sup>0</sup> state, relative to the total iron (e.g., Stone et al. 1982). This value has been routinely determined on all soil samples and many brecciated rocks since the early 1970s continuing on to today, virtually all measurements performed by Dr. Richard Morris at Johnson Space Center. The value of  $I_s/FeO$  has been determined to be largely a function of the abundance of agglutinitic particles in a soil, at least for the >45 μm fraction. Because the abundance of agglutinates is reasoned to increase with a soil’s exposure to space weathering at the lunar surface, this  $I_s/FeO$  value has been taken, for the <250 μm size fraction, as the maturity index for lunar soil. Immature soils have  $I_s/FeO$  values of 0–30, Submature from 30 to 60, Mature above 60, somewhat arbitrary divisions, but accepted by all lunar soil investigators (Morris 1976, 1978).

### Agglutinitic Glass versus Grain Size and Maturity

It has been demonstrated (Taylor et al. 1996) that for a given mare soil, the abundances of agglutinitic glass increase significantly with decreasing grain size, as evidenced by the  $I_s/FeO$  values that increase with decreasing grain size. Recall that the maturity index,  $I_s/FeO$ , is an indication of the amount of np-Fe<sup>0</sup>. As shown by Taylor et al. (2001a,b) in a study of selected mare soils and illustrated in Fig. 2, the percentage increase in agglutinitic glass, from larger grain sizes to the smaller size fractions, is only on the order of 10–15%, whereas the  $I_s/FeO$  changes by about 100%. That is, with a decrease in grain size, the change in agglutinitic glass content is relatively small compared with the change in  $I_s/FeO$ . This logically leads to the suggestion that the large increase in  $I_s/FeO$  is due to another source of nanophase Fe<sup>0</sup>, in addition to that in the agglutinitic glass.

Recent discoveries (e.g., Keller and McKay 1993, 1997; Keller et al. 1999, 2000) have been made of another major source of np-Fe<sup>0</sup> present in thin patinas (<1 μm) on the surfaces of many lunar soil particles. Most of this np-Fe<sup>0</sup> formed by deposi-

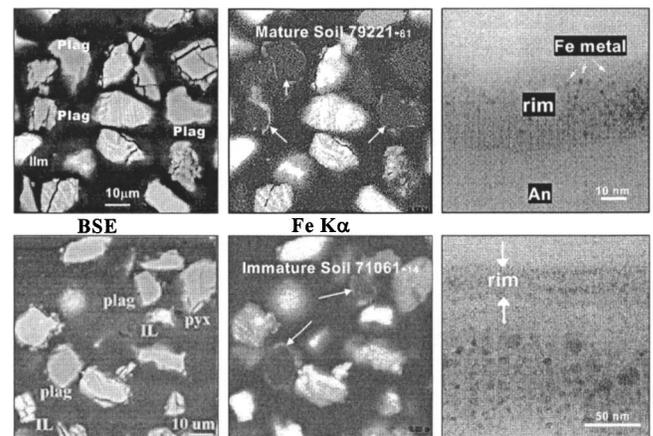


**Fig. 2.** Comparison of agglutinitic glass abundances with  $I_s/FeO$  for fine-grain size fractions of nine Apollo mare soils [after Taylor et al. (2000a,b)]

tion of vapor produced by abundant and intense micrometeorite impacts. A smaller portion may have formed by radiation damage (Bernatowicz et al. 1994). The exact mechanism of formation of the Fe<sup>0</sup>, although not well understood, was predicted long ago by Hapke et al. (1975).

### Surface-correlated Nanophase Fe<sup>0</sup>

The thesis on vapor-deposited patinas (e.g., Keller and McKay 1997; Pieters et al. 2000; Noble et al. 2001) has also found supporting evidence in recent studies (e.g., Taylor et al. 1999, 2000a,b, 2001a,b). The backscattered electron (BSE) and x-ray maps shown in Fig. 3 show that even plagioclase grains (CaAl<sub>2</sub>Si<sub>2</sub>O<sub>8</sub>–NaAlSi<sub>3</sub>O<sub>8</sub>) contain thin rims with appreciable Fe



**Fig. 3.** Backscattered electron images (BSE), Fe  $K\alpha$  x-ray maps, and TEM photos of particles from mare soils. The BSE image is a factor of the average atomic number of each phase; the Fe  $K\alpha$  x-ray map shows slight rims of Fe about the plagioclase grains, which do not contain appreciable Fe (<0.3%); and the TEM photos show the nature of the vapor-deposited rims of typical lunar grains that contain np-Fe<sup>0</sup> within a thin layer of SiO<sub>2</sub>-rich glass (Keller et al. 1999; Pieters et al. 2000; Taylor et al. 2000a,b). Note that the lower-right photo is that of an agglutinate where the differences in the relative sizes of the np-Fe<sup>0</sup> are clearly visible, with the vapor-deposited Fe<sup>0</sup> in the rim.

contents. The other mineral grains also contain Fe-rich rims, but the sensitivity of the Fe  $K\alpha$  map in Fig. 3 is not sufficient to show this. It would appear that *the presence of nanophase Fe<sup>0</sup> in the vapor-deposited patinas (rims) on virtually all grains of a mature soil provides the additional and abundant source for the greatly increased  $I_s/FeO$  values*, and as a side-note, causes the severe complications in the reflectance spectra of the Moon (Pieters and Taylor 1998; Pieters 1993; Pieters et al. 1998).

### Magnetic Separation of Lunar Soil Particles

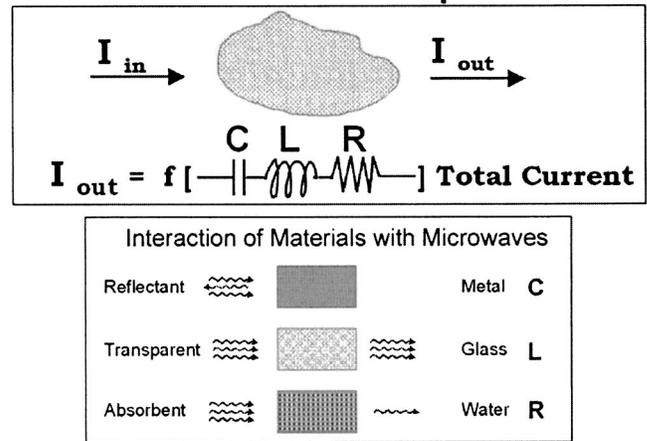
Several studies have been performed on lunar soils in order to determine the optimum conditions for the beneficiation of soil components for in situ resource utilization (ISRU) at a lunar base (Taylor and McKay 1992; Taylor et al. 1992, 1993; Chambers et al. 1994, 1995; Higgins et al. 1996). Using a Frantz Isodynamic Separator, specifically calibrated for susceptibility measurements, Taylor and Oder (1990) studied various size fractions of hi-Ti and low-Ti mare soils, as well as some from the highlands. They were able to successfully beneficiate the soil particles with decreasing efficiency as grain size decreased, down to 45–20  $\mu\text{m}$ . With sizes <20–40  $\mu\text{m}$ , they determined that separation was not possible. It appeared that “clumping” of these fine-sized grains was responsible. It was suspected that this fine-grained fraction was probably hydroscopic, causing the clumping. It is now apparent that this size fraction behaved as if virtually all the particles had relatively higher magnetic susceptibilities than the coarser particles. In retrospect, this behavior is now explainable, with our new knowledge, that each of these fine grains contains a surface patina of ferromagnetic nanophase Fe<sup>0</sup>.

### Physical Setting of the Nanophase Fe<sup>0</sup>

Recent experimentation with the <10  $\mu\text{m}$  fraction of mature hi-Ti mare soil 79221 (Taylor et al. 2000a,b) has shown that a strong hand magnet will easily attract practically all the grains, even those that are plagioclase, but have a thin patina of np-Fe<sup>0</sup>. This fine fraction, along with the high-magnetic-susceptibility agglutinitic glasses from coarser sizes, can be easily beneficiated from the lunar soil to make a feedstock for microwave processing. On a micro-scale, the np-Fe<sup>0</sup> both on the particle surfaces as well as in the agglutinitic glasses are effectively minute grains of a conductor each separated from the next by dielectric silicate glass, as discussed in detail in the following.

The presence of extensive amounts of np-Fe<sup>0</sup> on virtually all surfaces of soil grains is particularly advantageous where the grain-size of a soil fraction is small (e.g., <45  $\mu\text{m}$ ), since the surface to volume ratio is large. It will be possible to easily shape and form the soil and to sinter it slightly by “hot pressing.” The np-Fe<sup>0</sup> will ripen and grow during this process, thereby adding significantly to the adhesion and strength of the aggregates. In addition, the np-Fe<sup>0</sup> is located within a silicate glass, which being inherently unstable, will readily create additional fusion of the particles to each other. Indeed, as discussed in the following, the presence of this thin rim of np-Fe<sup>0</sup> on each grain can facilitate the formation of a fine-scale melt at the surface of these grains, thereby producing a glass binder—i.e., “a transient liquid-phase sintering.” The discovery of the abundance of this nanophase native Fe<sup>0</sup> on the surface of lunar soil grains provides unique properties to lunar soil that make it an ideal feedstock for microwave processing.

## Microwave Principles



**Fig. 4.** Interaction of microwave energy with materials as an analog to the movement of electrons in electric current. The sum of all losses of energy during microwave interactions with matter is called the “loss tangent.”

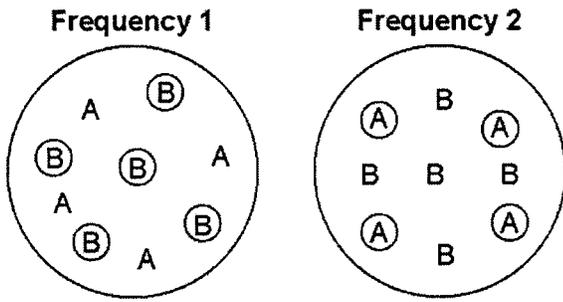
## Microwave Energy

### Principles

The microwave part of the electromagnetic spectrum corresponds to frequencies between 300 MHz and 300 GHz (Anderson and Leaver 1969; Ashby et al. 1986; Neas and Collins 1988; Sutton 1989; Smith 1996). However, most research and industrial activities involve microwaves only at 2.45 GHz and 915 MHz frequencies. Based on their microwave interaction, most materials can be classified into three categories—opaque, transparent, and absorbers. Bulk metals are opaque to microwave and are good reflectors (Fig. 4). Most other materials are either transparent or absorb microwaves to varying degrees at ambient temperatures, depending on the frequency of the microwave radiation.

As many homemakers have experienced, solid, nonporous metal readily reflects and effectively “shorts out” the microwaves. However, that same metal in a powdered form—effectively conductors separated by dielectric (particles and air)—will absorb microwaves at room temperature and will be heated effectively and rapidly (Roy et al. 1999). This principle has been patented (U.S. Patent No. 4,857,266), wherein Cu powder was lightly oxidized forming thin dielectric rims such that the conductive Cu grains were effectively separated by the dielectric CuO (U.S. Patent Nos. 4,880,578 and 4,892,581). Even when compressed into forms, the individual Cu cores of the grains are separated by the dielectric. It is primarily this *principle of metal separated by a dielectric glass* that will be expounded on in the following.

The heating that occurs in a microwave field depends on the local dielectric properties. For a simple monolithic material, the model is straightforward. However, as the number of phases increases, the model increases in complexity. Due to the differences in dielectric characteristics in a composite substance, each phase couples differently with the microwave energy thereby developing local temperature conditions (inhomogeneities) in the material. It is possible to have solid-state sintering of particles and development of local melt pockets, as well. Once a liquid phase is developed, then thermodynamic equilibrium and nucleation kinetics must be considered. Exactly how the electromagnetic field affects these thermo and kinetic factors becomes ever-more com-



**Fig. 5.** By varying the microwave frequency, energy can be coupled into different constituents of the materials. With the superposition of two frequencies, it is possible to effectively couple with different components of the lunar soil for more efficient heating.

plicated. But the combination of solid-state and liquid-phase sintering can result in better mechanical properties. This has been coined transient liquid-phase sintering.

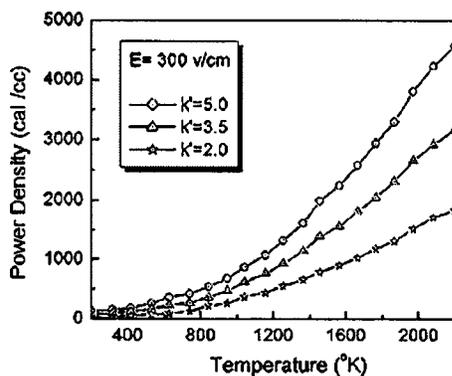
Specific frequencies “couple” with particular materials such that they cause substantial heating. In fact, as shown in Fig. 5, it is possible to have multiple microwave frequencies such that the energy can be coupled into different constituents of a given material. Such a situation is not simple to establish, especially for a multi-component material such as the lunar soil, but our preliminary modeling has shown that it is theoretically possible.

*Modeling* for the sintering of a dielectric in a microwave field involves the interactions of the electromagnetic radiation with matter and can be approximated by use of three basic components: *power density*; *half-power depth*; *heating rate*. These characterize how a material, such as a ceramic, will heat in an electromagnetic field. An initial analysis of these properties for the lunar soil was performed by Meek (1987) and Meek et al. 1986a, b). The power deposited per unit volume into a dielectric media by an electromagnetic field is

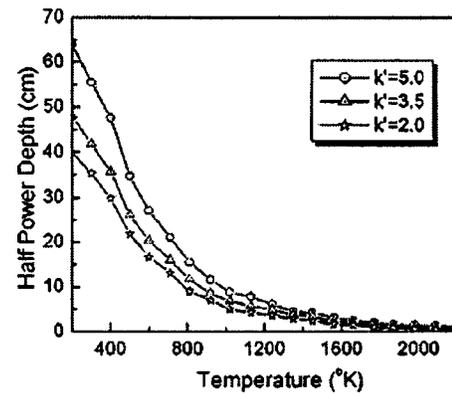
$$P = KfE^2k' \tan \delta$$

where  $P$ =power per unit volume;  $K$ =constant;  $f$ =frequency of radiation;  $E$ =electric-field intensity;  $k'$ =relative dielectric constant; and  $\tan \delta$ =loss tangent.

Dielectric properties and loss tangent data were obtained from the literature and used in construction of Figs. 6–9 (Bassett and Shackelford 1972; Strangway et al. 1972; Olhoef et al. 1973; Olhoef and Strangway 1975). Fig. 6 shows the power density



**Fig. 6.** Calculated power density versus temperature coupled into lunar regolith using 2.45 GHz electro-magnetic radiation with  $E=300$  V/cm and various values of  $k'$  (Meek et al. 1986b)



**Fig. 7.** Half-power depth of penetration in lunar regolith as a function of temperature and dielectric constant (Meek et al. 1986b)

deposited into lunar regolith for varying regolith dielectric constant and for an electric field intensity of 300 V/cm.

The depth to which microwave energy will penetrate a substance is referred to as the “skin depth” ( $D_S$ ) and is formulated as

$$D_S = 1/2\pi\mu cf$$

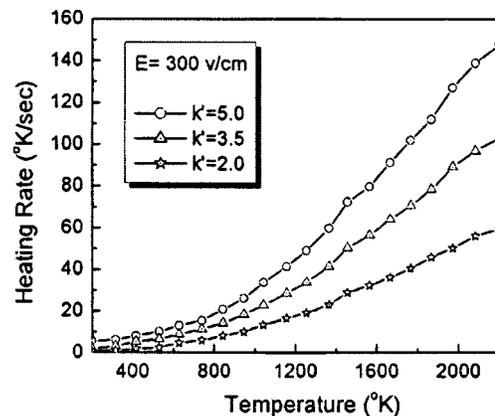
where  $D_S$ =depth (skin depth) of microwave energy penetration (centimeters);  $\mu$ =magnetic permeability;  $c$ =electrical conductivity; and  $f$ =frequency. With the case in point, the size of the metallic Fe grains as “suspended” within the glass is sufficiently small such that rather than reflecting the microwave energy, as large pieces of metallic Fe would, they react as small conductors with the absorption of the microwave energy.

The next expression allows us to determine the depth into the dielectric material where the power is half its value at the surface. This is called the half-power depth and is defined as

$$D_H = 3\lambda_0/[8.68\pi k' \tan \delta]$$

where  $D_H$ =half-power depth in centimeters;  $\lambda_0$ =wavelength of incident radiation;  $k'$ =relative dielectric constant; and  $\tan \delta$ =loss tangent. Fig. 7 shows how the half-power depth of penetration,  $D_H$ , varies as a function of regolith dielectric constant and temperature.

The last equation used in this modeling gives an approximate rate of temperature increase in a dielectric media that is heated in

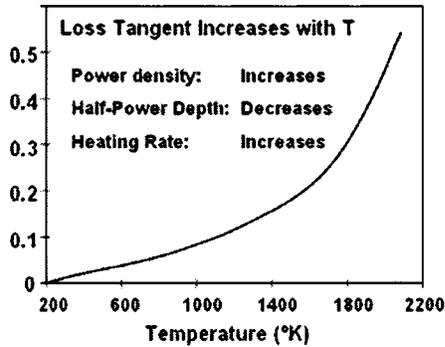


**Fig. 8.** Heating rate of typical lunar regolith with varying dielectric constant and varying temperature. Electric field intensity is 300 V/cm (Meek et al. 1986b).

## MICROWAVE HEATING:

IMPORTANT PARAMETERS FOR MATERIAL RESPONSE

DIELECTRIC CONSTANT,  $\epsilon_r$ , AND LOSS TANGENT,  $\tan \delta$



$\text{TAN } \delta = \text{Sum of All Losses from All Mechanisms during Microwave Heating}$

**Fig. 9.** The interaction of microwave radiation is such that the loss tangent increases logarithmically with temperature increase. The significant increase in the loss tangent results in the concentration of energy into ever smaller volumes of matter.

an electromagnetic field. This is only approximate because it does not include heat losses due to conduction, convection, radiation, or change of state

$$\Delta T \approx 8 \times 10^{-12} f E^2 k' \tan \delta / \rho C_p$$

where  $\Delta T$ =heating rate in  $^{\circ}\text{C}/\text{min}$ ;  $f$ =frequency;  $E$ =electric-field intensity;  $k'$ =relative dielectric constant;  $\tan \delta$ =loss tangent;  $\rho$ =density of material; and  $C_p$ =heat capacity. Fig. 8 shows how lunar regolith will heat in a microwave field as a function of soil dielectric properties in an applied electric field of 300 V/cm.

From this heating model, it is apparent that for an applied frequency of 2.45 GHz ( $\sim 10^{-6}$  eV photon energy) at 500 K and for a regolith with a  $k'$  of 5, the power density is approximately 250 cal/cc. Initially, heating is dominated by the applied electric field, “ $E$ .” Power density increases with the square of the electric-field intensity  $E$ . As temperature increases, a critical temperature is reached at which the dielectric loss  $\tan \delta$  becomes of primary importance in the ability to deposit energy into the material. As shown in Fig. 7, the amount of energy deposited per unit volume increases as temperature increases. Fig. 8, however, shows that as temperature increases, the half-power depth of penetration decreases. Initially at low temperature, 2.45 GHz microwave energy is easily deposited into a regolith depth of approximately 65 cm for a material dielectric constant of 3.5. At this depth, the power density is one-half that at the surface of the soil. As is apparent from Fig. 8, the half-power depth of penetration decreases as temperature increases. This decrease is again a function of  $\tan \delta$ . As  $\tan \delta$  increases,  $H$  decreases. The shape of the half-power depth curve is the inverse of the power density curve. In order to increase  $H$ , microwave radiation of a longer wavelength (lower frequency) should be used. Recognizing that low frequency (2.45 GHz) microwave heating occurs predominately as dielectric heating and then becomes dominated by the dielectric loss of the material, the rate of temperature rise starts off slowly, but increases at a rate that is influenced by the dielectric loss of the regolith. The shape of the heating curve is also influenced by the loss tangent of the soil. As regolith temperature increases, the volume of material being heated decreases due to the decrease in  $H$ . This is not of primary importance because the initial volume is

heated so rapidly that by the time the half-power depth decreases appreciably, the soil is already sintered. Fig. 9 shows the heating rate of lunar regolith as a function of soil dielectric constant and an applied electric field intensity of 300 V/cm. As regolith temperature increases, the rate of temperature rise  $\Delta T$  increases. At a regolith temperature of 1000 $^{\circ}\text{C}$ , the heating rate of a soil with a dielectric constant of 5 is approximately 50 $^{\circ}\text{C}/\text{s}$ . By increasing  $E$ , the heating rate can be markedly increased and sintering of the regolith can be accomplished very rapidly.

With a homogeneous distribution of two materials of contrasting dielectric constants in a composite, the lowest dielectric phase will have the highest electric field intensity. As sintering ensues, the density of this area increases in an attempt for the radiation to homogenize the dielectric constant. But as this occurs, more and more power is deposited into the lower-density regions with the lower dielectric phase. Another way to look at this is: Once dense regions begin to form, the microwave field will begin to decouple from these regions and couple more strongly to the regions of low density and low dielectric constant. The net goal of the microwave field is to create a material with a uniform dielectric constant.

### Dynamics of Microwave Heating

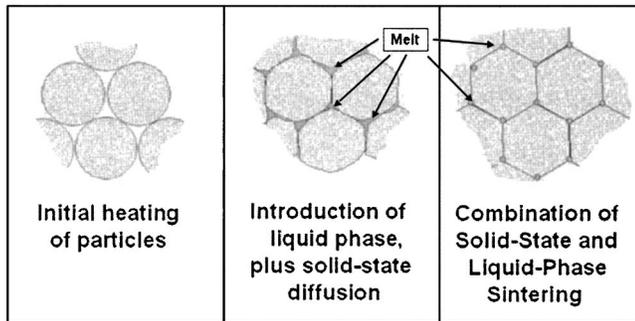
Inspection of the above-presented equations provides insight into the effects of microwave energy as temperature increases. Fig. 9 shows the effects of temperature on the “loss tangent” term,  $\tan \delta$ , that is of major importance here. In the equations for the power density and heating rate, the  $\tan \delta$  term is in the numerator, with the result that both the power density and heating rate increase dramatically as temperature increases; however, the half-power depth, where the loss tangent is in the denominator, decreases. These effects are at the center of the microwave heating, i.e., the power of the microwave energy becomes ever-more concentrated into a smaller volume as temperature increases. With this net effect, it is imperative that the frequency of radiation and the electric-field intensity be properly balanced such that the heating does go into a “run away mode,” which is not easily controlled.

### Experimentation with Lunar Simulants

As should be apparent from the above-presented discussion, there are no “lunar simulants” that have the specific properties of  $\text{np-Fe}^0$  such that they may be used for microwave heating and sintering experiments. Two major lunar simulants exist: MLS-1 and JSC-1. The Minnesota lunar simulant (MLS-1) consists of crushed high-Ti diabase (basalt) from Duluth that has been crushed so as to approximately duplicate the particle-size distribution of Apollo 11 lunar soil. Portions of this crushed rock have been melted to simulate the large glass components of lunar soil. However, it must be realized that this Minnesota rock was chosen for a simulant only because it had high-Ti content and general composition similar to Apollo 11 soil 10084. Its mineral chemistries were near that of the lunar phases, but they contain large amounts of  $\text{Fe}^{3+}$ , entirely atypical of lunar material. However, these lunar simulants have been studied extensively by microwave processing (e.g., Meek et al. 1988a,b,c; Nehls et al. 1989; Allen et al. 1992, 1994) with no unexpected results.

It is the large amounts of agglutinitic glass, typically  $>50\%$ , that make the lunar soil so unusual compared with any terrestrial analogs. This was the impetus that caused the Johnson Space Center to establish lunar simulant JSC-1. This is a crushed volca-

## Sintering Progress of Powder Particles By Microwave Energy



**Fig. 10.** Diagrammatic representation of the progressive sintering of lunar soil (left to right). It is the development of the “transient liquid phase” that is promoted by the surficial nanophase  $\text{Fe}^0$ . It is evident that this coupling with the microwave radiation creates temperatures far in excess of that present in the centers of the minerals.

nic tuff that consists of a large percentage of glass shards and angular grains that more closely approximates the physical abrasive properties of lunar soil, but not its chemistry. Thus, the two main lunar simulants used by scientists and engineers worldwide only have some particular properties which are similar to lunar soil. This is not widely appreciated. However, there is presently no lunar soil simulant that has the nanophase  $\text{Fe}^0$  properties that make experimentation with microwave radiation realistic to any degree. However, we are fortunate to have been allocated some Apollo 17 soil from NASA.

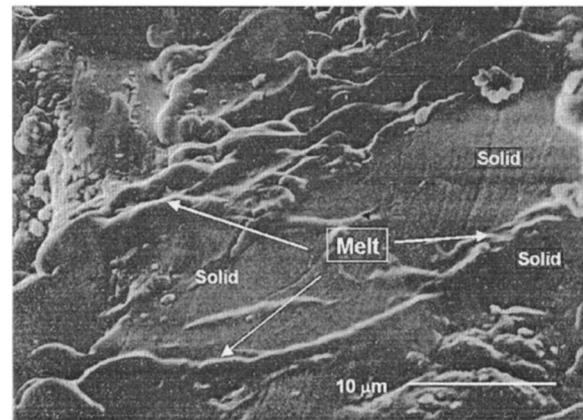
### Sintering of Lunar Simulants

Microwave heating experiments of lunar soil, conducted by Meek and colleagues (Meek et al. 1986a,b; Vaniman et al. 1986), were found to be extremely difficult to decipher because they did not consider the presence of the abundant nanophase  $\text{Fe}^0$ . Instead, they attempted to interpret their sintering experiments in light of solar-wind radiation defects on the surfaces of the grains. They also speculated that the mineral ilmenite ( $\text{FeTiO}_3$ ), abundant in some of the mare soils, was causing some of the heating (Wright et al. 1989). But the distinct differences in microwave effects between the actual lunar soil and “apparently representative” lunar simulants were not understood. As presented in the following, we now appreciate the cause for the unique behavior of lunar soil with microwave radiation, and it cannot be emphasized enough that this is an unusual property for any naturally occurring material. Basically, the minute sizes of the nanophase metallic Fe is small enough such as to be less than the skin depth of the microwave energy. This makes each of the metallic Fe grains into a conductor [versus typical reflector], separated from the other metallic Fe particles by the dielectric glass. The conductor abilities of the metallic Fe act as an absorber of the microwave energy, thereby creating “energy sinks” with the effective generation of large quantities of heat.

### Experimentation with Real Lunar Soil

Experimentation on microwave sintering and melting of lunar soil involves several complex considerations. Our furnace was designed for the use of either microwave radiation alone or the

## Microwave Melting Along Grain Boundaries of Mare Soil



**Fig. 11.** Scanning electron microscopy photo of the surface of lunar soil grains after sintering with 2.45 GHz microwave radiation. Note the presence of a melt phase (now glass) along grain boundaries. Although the “measured temperature” of the charge was  $<900^\circ\text{C}$ , the temperature necessary to produce the melt along the grain boundaries was  $>1,200^\circ\text{C}$ .

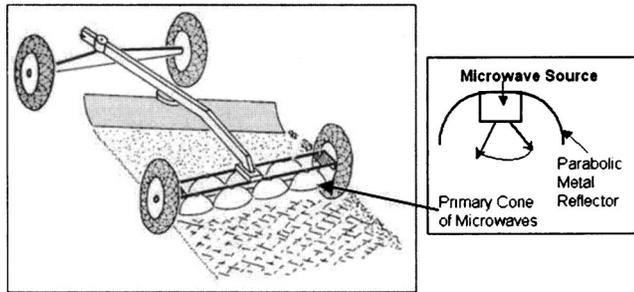
hybrid heating using the addition of conventional heating elements as well. One of the major problems in microwave experiments is determining the real temperature of the sample (German 1996). An average temperature of a material can be obtained by an optical pyrometer or use of shielded thermocouples. But, as demonstrated by Meek et al. (1991), temperature gradients on the orders of hundreds of degrees can exist on a micro-scale. In fact, this is thought to be the factor causing the production of a “transient liquid phase” during sintering, as depicted schematically in Fig. 10 (Meek et al. 1987; Rahaman 2003). This production of the “transient liquid phase” is well demonstrated by a photomicrograph of one of our experiments with Apollo lunar soil, as shown in Fig. 11. Note the melt (now glass) formed along grain boundaries. In addition, the actual shape of the body being radiated affects the local heating rates significantly; the microwave frequency can play a major role in the production of temperature gradients that develop in the body; therefore, achieving sufficiently uniform heating is not straightforward. This is further complicated by the multi-phase nature of the lunar soil.

*Microwave versus conventional heating:* Due largely to the presence of np- $\text{Fe}^0$  in lunar soil, the advantages of using microwave radiation for processing are immense. A comparison against normal conventional heating (large resistance-driven) shows that microwave heating of lunar soil has many distinct perks. Rapid heating rates [ $1000^\circ/\text{min}$ ] to high temperatures [ $2000^\circ\text{C}$ ], enhanced reaction rates [faster diffusion rates], with faster sintering kinetics [shorter sintering times] and lower sintering temperatures [energy savings], tailored microstructures [improved mechanical properties], considerably reduced processing time, and process simplicity, are all factors leading to the bottom line which is that of tremendous energy savings. Granted, the efficiency of electrical creation of microwave energy is probably only about 50%, but this initial loss is more than compensated for by the extreme microwave coupling with the nanophase metallic Fe. It is estimated that the total energy savings of microwave heating versus conventional heating approaches 100%, with the value-added factors of heating time and “inside-out” heating with microwaves.

## LUNAR SOIL PROCESSING & PRODUCTS

### SINTERING and MELTING

#### Creating Smooth-Sintered to Glassy Surfaces on the Moon



**Fig. 12.** Cartoon of a “Lunar Road-Paving Wagon” with two sets of magnetrons that can be set to various frequencies and power in order to effectively sinter/melt the lunar soil, thereby constructing a trafficable road (US 101)

This latter property can be of exceptional value because of the efficiency of microwave heating facility design.

### Products from Microwave Processing of Lunar Soil

It is not difficult to conceive of applications of microwave processing of lunar soil for a multitude of products. Fig. 12 depicts “a road-paving wagon” to be pulled over the lunar surface, consisting of a front blade for minor smoothing of the regolith, with two rows of magnetrons in the back. The first row of magnetrons is set in power and frequency such that the half-power depth will assure that the soil beneath the wagon will be solidly sintered to a depth of 0.5 m. The second row is set such that the magnetrons will completely melt the uppermost 3–5 cm of the soil, which will crystallize to glass as the wagon passes on. In addition, as this wagon passes along it will heat up and thereby *release most of the solar-wind particles* within the soil, most notably hydrogen, helium, carbon, and nitrogen. An apparatus should obviously be constructed on here in order to capture these valuable elements. As emphasized by Taylor and Kulcinski (1999), the value of helium-3 for the generation of clean, non-radioactive fusion energy may well replace the loss of fossil-fuel electricity in the future.

Sintering of pre-formed blocks of soil can be used to form *solid bricks* for various construction purposes (e.g., igloos). An impact crater might be selected and smoothed out to a parabolic shape. Subsequent microwave treatment of the surface could produce an *antenna dish*, complete with a smooth glass surface. Or, this dish might be cut into sections for movement and reassembly to another location—e.g., a station at L<sub>1</sub> orbit? Processing of pre-molded soil can produce *strong structural components*. Melting of the mare low-viscosity soil for *blowing glass wool* or *pulling of glass fibers*. The variety of possible applications awaits only an *active imagination*.

### Conclusion

The lunar soil is the best material for immediate use for ISRU purposes in the immediate vicinity of and on the Moon. However,

studying the precise properties of the fine fraction of the lunar soil, the portion of particular interest here, is not possible with any known lunar soil analog (e.g., MS-1, JS-1, etc.). Each simulant has properties “like” but not identical to the lunar soil. In particular, the ubiquitous presence of nanophase Fe<sup>0</sup> (3–10 nm) in the glass of the soil and present on the surfaces of almost all grains is such a fine-scale feature that it is virtually impossible to duplicate exactly with a simulant. Fortunately, we have the necessary lunar soil (with NASA permission for ISRU purposes) to investigate the coupling of nanophase elemental Fe in and on the soil particles and have performed preliminary experiments with the soil. The 80 g of Apollo 17 soil that has been allocated to the senior author, Larry Taylor, is the only real feedstock with which to study the microwave sintering of lunar soil. This has been amply indicated in the discussion above of the unique properties of the lunar soil that make all lunar soil simulants insufficient for the proposed studies.

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### References

- Allen, C. C., Graf, J. C., and McKay, D. S. (1994). “Sintering bricks on the Moon.” *Engineering, construction, and operation in space III*, ASCE, Reston, Va., 1220–1229.
- Allen, C. C., Hines, J. A., McKay, D. S., and Morris, R. V. (1992). “Sintering of lunar glass and basalt.” *Engineering, construction, and operation in space III*, ASCE, Reston, Va., 1209–1218.
- Anderson, J. C., and Leaver, K. D. (1969). *Materials science*, Van Nostrand Reinhold, New York, 1–15.
- Ashby, M. F., and Jones, J. J., Larry, D. R. H. (1986). “The mechanical properties of ceramics.” *Engineering materials 2. An introduction to microstructures, processing and design*, Pergamon, Tarrytown, N.Y., 162–182.
- Bassett, H. L., and Shackelford, R. G. (1972). “Dielectric properties of Apollo 14 lunar samples at microwave and millimeter wavelengths.” *Proc., 3rd Lunar Sci. Conf., Geochim. Cosmochim. Acta*, 3, 3157–3160.
- Bernatowicz, T. J., Nichols, R. H., Jr., and Hohenburg, C. M. (1994). “Origin of amorphous rims on lunar soil grains.” *Lunar Planet. Sci. Conf., XXV*, Lunar and Planetary Institute, Houston, 105–106.
- Carrier, W. D., III. (1991). “Geotechnical properties of lunar regolith.” *Lunar sourcebook*, Heiken, G., Vaniman, D., French, B., eds., Cambridge University Press, New York, 475–530 and 567–580, 585–594.
- Carrier, W. D., III. (2003). “Particle size distribution of lunar soil.” *ASME J. Eng. Mater. Technol.*, 129(10), 956–959.
- Chambers, J. G., Taylor, L. A., Patchen, A., and McKay, D. S. (1994). “Mineral liberation and beneficiation of lunar high-Ti mare basalt, 71055: Digital-imaging analyses.” *Engineering, construction, and operations in space IV*, ASCE, New York, 878–888.
- Chambers, J. G., Taylor, L. A., Patchen, A., and McKay, D. S. (1995). “Quantitative mineralogical characterization of lunar high-Ti mare ba-

- salts and soils for oxygen production." *J. Geophys. Res., [Planets]*, Vol. 100, 14,391–14,401.
- German, R. M. (1996). *Sintering theory and practice*, Wiley, New York, 550.
- Hapke, B., Cassidy, W. A., and Wells, E. N. (1975). "Effects of vapor-phase deposition processes on the optical, chemical, and magnetic properties of the lunar regolith." *Moon*, 13, 339–353.
- Higgins, S. J., Taylor, L. A., Patchen, A., Chambers, J. G., and McKay, D. S. (1996). "X-ray digital-imaging petrography: Technique development for lunar mare soils." *Meteorit. Planet. Sci.*, 34, 125–138.
- Housley, R. M., Grant, R. W., and Paton, N. E. (1973). "Origin and characteristics of excess Fe metal in lunar glass welded aggregates." *Proc., 4th Lunar Sci. Conf.*, 2737–2749.
- Hu, H. N., and Taylor, L. A. (1977). "Lack of chemical fractionation in major and minor elements during agglutinate formation." *Proc., 8th Lunar Planet. Sci. Conf.*, Vol. 3, *Geochemical et Cosmochimica Acta*, Suppl 8, Pergamon, New York, 3645–3656.
- Keller, L. P., et al. (1999). "Space weathering alteration of lunar soil grains." *Lunar Planet. Sci. XXX*, (CD-ROM 1820), Lunar and Planetary Institute, Houston.
- Keller, L. P., et al. (2000). "Space weathering in the fine size fraction of lunar soils: Mare/highland differences." *Lunar Planetary Sci. Conf. XXXI*, (CD-ROM 1655), Lunar and Planetary Institute, Houston.
- Keller, L. P., and McKay, D. S. (1993). "Discovery of vapor deposits in the lunar regolith." *Science*, 261, 1305–1307.
- Keller, L. P., and McKay, D. S. (1997). "Nature and origin of rims on lunar soil grains." *Geochim. Cosmochim. Acta* 61, 2331–2340.
- Meek, T. T. (1987). "A proposed model for the sintering of a dielectric in a microwave field." *J. Mater. Sci. Lett.*, 6, 638–640.
- Meek, T. T., Brooks, M., Blake, R. D., Bradbury, J., and Katz, J. (1988a). "Cation diffusion in glass heated in a microwave field." *J. Mater. Sci. Lett.*, 7, 928–931.
- Meek, T. T., Fayweather, M. J., Godbole, M. J., Vaniman, D. T., and Honnell, R. (1988b). "Sintering lunar simulants using 2.45 GHz radiation." *Engineering, construction, and operations in space*, ASCE, Reston, Va., 102–110.
- Meek, T. T., Vaniman, D. T., Blake, R. D., and Cocks, F. H. (1986a). "Electromagnetic energy applied to and gained from lunar materials." *Symposium 86, The first lunar development symposium*, Maglev, Atlantic City, N.J., 40–42.
- Meek, T. T., Vaniman, D. T., Blake, R. D., and Godbole, M. J. (1987). "Sintering of lunar soil simulants using 2.45 GHz microwave radiation." *Lunar and planetary science XVIII*, The Lunar and Planetary Institute, Houston, 635–636.
- Meek, T. T., Vaniman, D. T., Cocks, F. H., and Wright, R. A. (1986b). "Microwave processing of lunar materials: Potential applications." *Lunar bases and space activities in the 21st century*, NASA, Washington, D.C., 479–486.
- Meek, T. T., Vaniman, D. T., Fayweather, L. A., and Godbole, M. J. (1988c). "Processing lunar simulants using 2.45 GHz microwave radiation." *Engineering, construction, and operations in space*, ASCE, Reston, Va., 102–110.
- Meek, T. T., Zhang, X., and Rader, M. (1991). "An analysis of the individual grain structure of an oxide heated using microwave radiation and heated conventionally." *Ceram. Trans.*, 21, 81–93.
- Morris, R. V. (1976). "Surface exposure indices of lunar soils: A comparative FMR study." *Proc., 7th Lunar Planet. Sci. Conf.*, 315–335.
- Morris, R. V. (1978). "The surface exposure (mature) of lunar soils: Some concepts and  $Is/FeO$  compilation." *Proc., 9th Lunar Planet. Conf.*, Vol. 2, *Geochemical et Cosmochimica Acta*, Suppl. 9, Pergamon, New York, 2287–2297.
- Neas, E. D., and Collins, M. J. (1988). "Microwave heating: Theoretical concepts and equipment design." *Introduction to microwave sample preparation*, H. M. Kingston and L. B. Jassie, eds., American Chemical Society Professional Reference Book, Washington, D.C., 7–32.
- Nehls, M. K., Park, S. S., and Meek, T. T. (1989). "Processing lunar simulant materials using 2.45 GHz radiation." *Proc., 8th Biennial Conf. Space Manufacturing*, Space Studies Institute, Princeton Press, Princeton, N.J., 94–96.
- Noble, S. K., et al. (2001). "The optical properties of the finest fraction of lunar soil: Implications for space weathering." *Meteorit. Planet. Sci.*, 36, 31–42.
- Olhoeft, G. R., and Strangway, D. W. (1975). "Dielectric properties of the first 100 meters of the Moon." *Earth Planet. Sci. Lett.*, 24, 394–404.
- Olhoeft, G. R., Strangway, D. W., and Frisillo, A. L. (1973). "Lunar sample electrical properties." *Geochim. Cosmochim. Acta*, 3, 3133–3149.
- Pieters, C. M. (1993). "Compositional diversity and stratigraphy of the lunar crust derived from reflectance spectroscopy." *Remote geochemical analysis: Elemental and mineralogical composition*, C. M. Pieters and P. A. J. Englert, eds., Cambridge University Press, New York, 309–339.
- Pieters, C. M., and Taylor, L. A. (1998). "Visible to mid-infrared spectra of lunar mare soils." *Lunar Planet. Sci. XXIX*, (CD-ROM), Lunar and Planetary Institute, Houston.
- Pieters, C. M., Taylor, L. A., Noble, S. K., Keller, P., Hapke, B., Morris, R. V., Allen, C. C., and Wentworth, S. (2000). "Space weathering on airless bodies: Resolving a mystery with lunar samples." *Meteorit. Planet. Sci.*, 35, 1101–1107.
- Pieters, C. M., Taylor, L. A., Takahiro, H., McKay, D., Wentworth, S., and Morris, R. (1998). "Optical properties and abundances of minerals and glasses in the 10 to 44 micron size fraction of mare soils. Part II." *Lunar Planet. Sci. XXIX*, (CD-ROM).
- Rahaman, M. N. (2003). *Ceramic processing and sintering—Sec. Ed.*, Dekker, New York, 875.
- Roy, R., Agrawal, D., Cheng, J., and Gedevisanishvii, S. (1999). "Full sintering of powdered-metal bodies in a microwave field." *Nature (London)*, 339, 668–670.
- Stone, C., Taylor, L. A., McKay, D. S., and Morris, R. V. (1982). "Ferromagnetic resonance intensity ( $I_r$ ): A rapidly determined indicator of lunar glass bead origin." *J. Geophys. Res.*, 87, A182–196.
- Strangway, D. W., Chapman, W. B., Olhoeft, G. R., and Carnes, J. (1972). "Electrical properties of lunar soil dependence upon frequency, temperature, and moisture." *Earth Planet. Sci. Lett.*, 16, 275–281.
- Sutton, W. H. (1989). "Microwave processing of ceramic materials." *Am. Ceram. Soc. Bull.*, 68, 376–386.
- Taylor, L. A. (1988). "Generation of native Fe in lunar soil." *Engineering, construction, and operations in space I*, ASCE, New York, 67–77.
- Taylor, L. A., et al. (1992). "Magnetic beneficiation of high-Ti mare basalts: Petrographic analysis." *Lunar Planet. Sci. Conf. XXIII*, Lunar and Planetary Institute, Houston, 1415–1416.
- Taylor, L. A., et al. (1993). "Evaluation of lunar rocks and soils for resource utilization: Detailed image analysis of raw material and beneficiated products." *Lunar Planet. Sci. Conf. XXIV*, Lunar and Planetary Institute, Houston, 1409–1410.
- Taylor, L. A., et al. (1999). "Integration of the chemical and mineralogical characteristics of lunar soils with reflectance spectroscopy." *Proc., Lunar Planet. Sci. Conf.*, (CD-ROM), 1859, Lunar and Planetary Institute, Houston.
- Taylor, L. A., et al. (2000a). "Major contributions to spectral reflectance opacity by non-agglutinitic, surface-correlated nanophase iron." *Proc. Lunar Planet. Sci. Conf.*, (CD-ROM), abstract 1842, Lunar and Planetary Institute, Houston.
- Taylor, L. A., et al. (2001a). "The effects of space weathering on Apollo 17 mare soils: Petrographic and chemical characterization." *Meteorit. Planet. Sci.*, 36, 285–289.
- Taylor, L. A., et al. (2001b). "Space weathering of lunar mare soils: New understanding of the effects on reflectance spectroscopy." *Proc. of Space 2000, Engineering, constructions, operations, and business in space*, S. Johnson, et al., eds., ASCE, Reston, Va., 703–711.
- Taylor, L. A., and Cirlin, E.-H. (1995). "A review of ESR studies on lunar

- samples." *IONICS—ESR dating and dosimetry*, IONICS, Tokyo, 19–39.
- Taylor, L. A., and Kulcinski, G. L. (1999). "Helium-3 on the Moon for fusion energy: The Persian Gulf of the 21st Century." *Solar System Research*, 33, 338–345 (English translation); from (1999). *Astronomischeskiizz*, No. 5, 386–394.
- Taylor, L. A., and McKay, D. S. (1992). "An ilmenite feedstock on the Moon: Beneficiation of rocks versus soils." *Lunar Planet. Sci. Conf. XXIII*, Lunar and Planetary Institute, Houston, 1411–1412.
- Taylor, L. A., and Meek, T. T. (2004). "Microwave processing of lunar soil." *Proc., Intro. Lunar Conf. & Int. Lunar Explor. Work. Grp 5, Adv. Astronaut. Sci.*, 108, 109–123.
- Taylor, L. A., and Oder, R. R. (1990). "Magnetic beneficiation of high-land and high-Ti mare soils: Rock, mineral, and glassy components." *Engineering, constructions, and operations in space II*, ASCE, New York, 143–152.
- Taylor, L. A., Patchen, A., Taylor, D.-H., Chambers, J. G., and McKay, D. S. (1996). "X-ray digital imaging and petrography of lunar mare soils: Data input for remote sensing calibrations." *Icarus* 124, 500–512.
- Taylor, L. A., Pieters, C. M., Keller, L. P., Morris, R. V., and McKay, D. S. (2001b). "Lunar mare soils: Space weathering and the major effects of surface-correlated nanophase Fe." *Geophys. Res.*, 106, 27,985–27,999.
- Vaniman, D. T., Meek, T. T., and Blake, R. D. (1986). "Fusing lunar materials with microwave energy. II. Melting of a glassy Apollo 11 soil stimulant." *Lunar and Planetary Science XVII*, The Lunar and Planetary Institute, Houston, 911–912.
- Wright, R. A., Cocks, F. H., Vaniman, D. T., Blake, R. D., and Meek, T. T. (1989). "Thermal processing of ilmenite and titania-doped hematite using microwave energy." *J. Mater. Sci.*, 24, 1337–1342.