

ELECTROSTATIC CONCENTRATION OF LUNAR SOIL MINERALS

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Estimates of the magnetic susceptibility of lunar ilmenite indicate that electrostatic separation may be preferable to magnetic separation as a beneficiation technique for concentration of the mineral in lunar soil. Single-pass electrostatic separations of terrestrial ilmenite from a lunar soil simulant in the 0.15–0.09 mm size range on a slide-type separator yielded higher grades in air or nitrogen (89 and 90 wt %, respectively) than in vacuum (78 wt %) due to the effects of air resistance, charging of the feed by gas ions, and the absence of vibratory feed in the vacuum runs. Single-pass concentrations on a 0.09–0.15 mm fraction of Apollo 11 soil 10084,853 increased the lunar ilmenite grade from 7 wt % to 51 wt % in dry nitrogen with the ferromagnetic agglutinates removed and to 29 wt % in vacuum (10^{-5} torr) with all agglutinates present. If all ilmenite-bearing soil phases are included, one-pass grades of lunar ilmenite increase to the mid 60s for the N₂ runs and to the mid 30s for the vacuum run. Soil ilmenite behaved like a conductor or semi-conductor and the agglutinates like non-conductors in all lunar runs. This behavior suggests that ilmenite grades and recoveries in the high 90s can be expected from comparable mare soil fractions with fully liberated ilmenite using multistage-multipass electrostatic concentrators of commercial design. However, redesign of the separator geometry is required for optimal performance in vacuum.

INTRODUCTION

Numerous authors (Andrews and Snow, 1981; Waldron and Criswell, 1982; Davis, 1983) have projected substantial economic advantages for lunar oxygen production as a major fuel and gas resource in extended space industrial operations. Several systems studies of lunar oxygen production (Davis, 1983; Gibson and Knudsen, 1985) have referenced a process utilizing ilmenite, the most abundant oxide in the returned lunar samples. Lunar ilmenite consists of 47 wt % FeO and 53 wt % TiO₂ stoichiometrically and is most heavily concentrated in the maria (McKay and Williams, 1979). Williams and Mullins (1983) have demonstrated the feasibility of oxygen extraction from terrestrial ilmenite by hydrogen reduction of the FeO component.

The paramagnetic properties of ilmenite at ordinary temperatures suggest magnetic separation as a method for concentrating it from mare soil. Accordingly, the magnetic susceptibility of lunar ilmenite was estimated in a 0.09–0.15 mm split of Apollo 11 soil 10084 using a Franz model L1 magnetic separator at the NASA/Johnson Space Center (JSC). The susceptibility was approximately 76×10^{-6} cgs mass units, similar to synthetic stoichiometric terrestrial ilmenite but orders of magnitude below that of natural terrestrial ilmenite, which averages $26,800 \times 10^{-6}$ cgs mass units (Carmichael, 1982). The low lunar ilmenite susceptibility probably reflects the virtual absence of Fe³⁺ compared with terrestrial varieties.

The susceptibility determination was made on the soil split after the high susceptibility agglutinates (approximately 75% of total sample agglutinates by microscopic count) were removed using a permanent magnet with a strength of approximately 1500 gauss. When the balance of the sample was run on the Franz separator, all paramagnetic splits that contained ilmenite also contained comparable abundances of agglutinates that had not been removed by the magnet. It was concluded that the lunar ilmenite may be difficult to separate magnetically from the population of soil agglutinates with comparable susceptibilities. Therefore, the alternate technique of electrostatic concentration of lunar soil ilmenite has been studied, and experimental results using electrostatic separators are reported here. The separators are based on industrial designs and were fabricated by Lockheed Engineering and Management Services Co. at NASA/JSC.

MINERAL ELECTROSTATIC SEPARATION

The primary commercial application of mineral electrostatic beneficiation is in processing beach sands and alluvial deposits for titanium minerals. All of the heavy mineral beach sand plants in Australia, and most in the United States (Florida), use electrostatic methods to separate rutile and ilmenite from zircon and monazite (Fraas, 1962; Kelly and Spottiswood, 1982). The most common electrostatic separator designs use a drum or slide configuration. In both designs, a high intensity electric field (several kV/cm) is established by a high voltage electrode spaced a few cm from the grounded drum or slide. Many separators use an additional ionizing electrode above the field electrode to charge the mineral feed with air ions and electrons before it enters the accelerating field (Fraas, 1962; Carpenter, 1970; Moore, 1973). The slide design (Figs. 1 and 2) was used for these experiments because its performance was superior to the drum design for small samples (0.1–5.0 g) (Agosto, 1983) and because it is simpler to operate in vacuum.

Minerals falling through the separating field commonly acquire charge by one or some combination of the following mechanisms: (1) Electrostatic induction; (2) Contact charging; (3) Ionic charging.

Electrostatic induction occurs primarily in conducting and semi-conducting grains which, because they are grounded by the slide, acquire charge opposite in polarity to the field electrode and are pulled toward it. As a result, conductor and semi-conductor particles preferentially report to the conductor bin or bins farthest from the foot of the slide.

Contact charging occurs when a material with a lower electronic work function gives up electrons and becomes positively charged by contact with a higher work function material. Since the rate of charging and discharging is an exponential function of the particle surface conductivity (Kelly and Spottiswood, 1982; Inculet, 1982), conductors lose excess charge rapidly on the slide. Given time to acquire sufficient contact charge, non-conductors tend to retain the charge and move toward the electrode of opposite polarity. Grains that acquire insufficient charge fall into the non-conductor bin or bins at the foot of the slide.

Mineral charging also occurs in the separator due to gas ion and electron bombardment generated by the ambient electric field. This mechanism was not introduced intentionally in these experiments, but evidence for the presence of ionic charging was noted in separation runs made in dry nitrogen.

The charging behavior of terrestrial varieties of the most common mare soil minerals (anorthite, ilmenite, olivine, pyroxene) in four size ranges <0.5 mm has been studied in slide-type electrostatic separators (Agosto, 1983). Ilmenite electrostatic concentration in air for soil-analog size fractions below 0.09 mm was found to be substantially poorer than in the larger size fractions. The reduced performance resulted from partial clumping of fines on the slide as well as billowing and dispersal of the falling fines, which were substantially retarded by the air. Virtually every investigator has reported these effects for particles under 0.075 mm (Fraas, 1962; Carta *et al.*, 1964; Carpenter, 1970; Inculet, 1979; Kelly and Spottiswood, 1982). Since mean grain size for most lunar soils is under 0.1 mm, it appeared that vacuum operation of the separator might improve performance by eliminating the disruptive effects of air resistance on the flow of fines.

ILMENITE ELECTROSTATIC CONCENTRATION IN LUNAR SOIL SIMULANTS

Lunar soil simulants were prepared from mixtures of comminuted terrestrial anorthite (An_{90}), ilmenite (Quebec), olivine (Fe_{90}), and augite pyroxene ($Wo_{50}En_{33}$) in the weight ratio 4:1:1:4, respectively. Two size ranges (0.09–0.15 mm and 0.15–0.25 mm) were tested in air, and one size range (0.09–0.15 mm) was tested in nitrogen and in a vacuum of approximately 10^{-5} torr. Samples were weighed in quantities of 1–5 g and were washed and boiled in isopropyl alcohol until the supernatant fluid appeared clear. Prior to separation, all samples were dried, first on filter paper and then in glass receptacles in an air oven at 120° – 130° C overnight.

Ilmenite electrostatic separation grades and recoveries from the simulants are reported in each of three ambients: air at one atmosphere; dry nitrogen at about one torr positive pressure; and in vacuum at 10^{-5} ($\pm 20\%$) torr. In all cases, grade is defined to be the cumulative concentration (wt %) of ilmenite in the conductor bins; recovery is the wt % of total sample ilmenite reporting to those bins. Ilmenite concentration was determined in each bin by microscopic count. The wt % of ilmenite was then calculated by weighing the contents of each bin and assuming that the ilmenite fraction had a density ratio of 1.5 relative to the balance of the simulant sample.

Samples were maintained at 100° – 200° C in the separator prior to and during separation by heating both the vibratory feed hopper and the grounded slide to drive off adsorbed feed moisture and to enhance the conductivity of the ilmenite. Feed heating is a common practice in commercial electrostatic mineral separation operations (Fraas, 1962; Kelly and Spottiswood, 1982). Feed rate was approximately 6 g/min, and feed size ranged from a maximum of a few grams in the bench top runs in air to a few hundred milligrams in the nitrogen and vacuum runs.

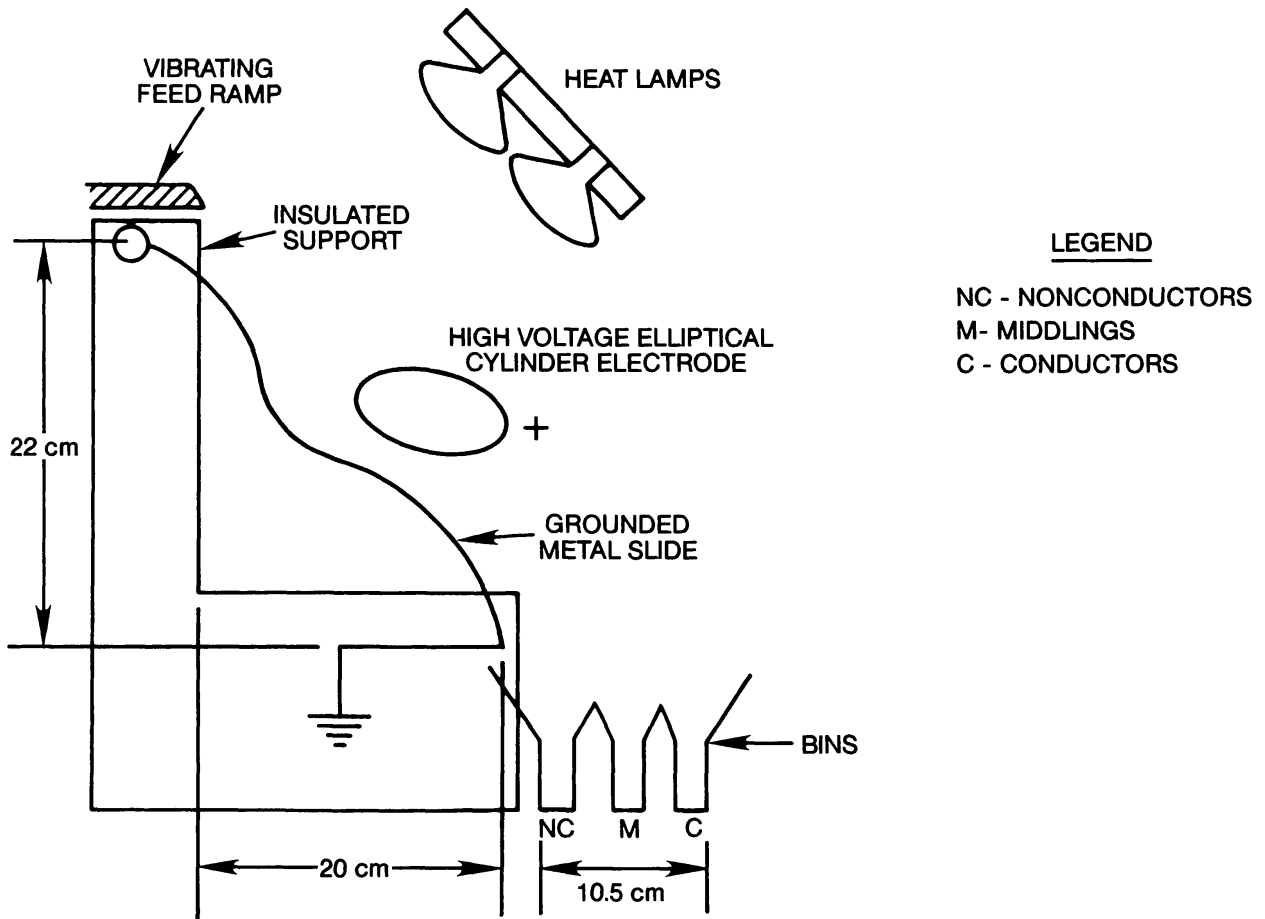


Figure 1. Mineral electrostatic separator: bench top slide configuration.

Maximum electric field strength was nominally +5 kV/cm DC but ranged between +2.5 and +7 kV/cm. Positive field electrode polarity was used throughout to make the ilmenite induced charge additive to the negative ilmenite contact charge on the aluminum slide (Agosto, 1984).

Four runs of ilmenite electrostatic concentration from simulants were made in air in a bench top apparatus (Fig. 1) where the feed hopper and slide were heated with infrared lamps. Separates were collected in three bins each 3.5 cm wide with bin 3 being the only conductor bin in the bench top setup. The feed size range was 0.15–0.25 mm.

Three other simulant runs in air and all simulant runs in N₂ and in vacuum were made in the slide configuration designed to fit the vacuum system (Figs. 2 and 3). The feed size range was 0.09–0.15 mm. The hopper and slide were resistance heated, and separates were collected in 7 bins each 2 cm wide. (The number of bins was increased to improve separates resolution for subsequent vacuum runs.) High concentrations of ilmenite reported to bins 2 through 7. Mean grade for all runs in air after one pass was 89±6 wt % (up from a mean ilmenite starting concentration of 8.8 wt %), and recovery was 51±9 wt %.

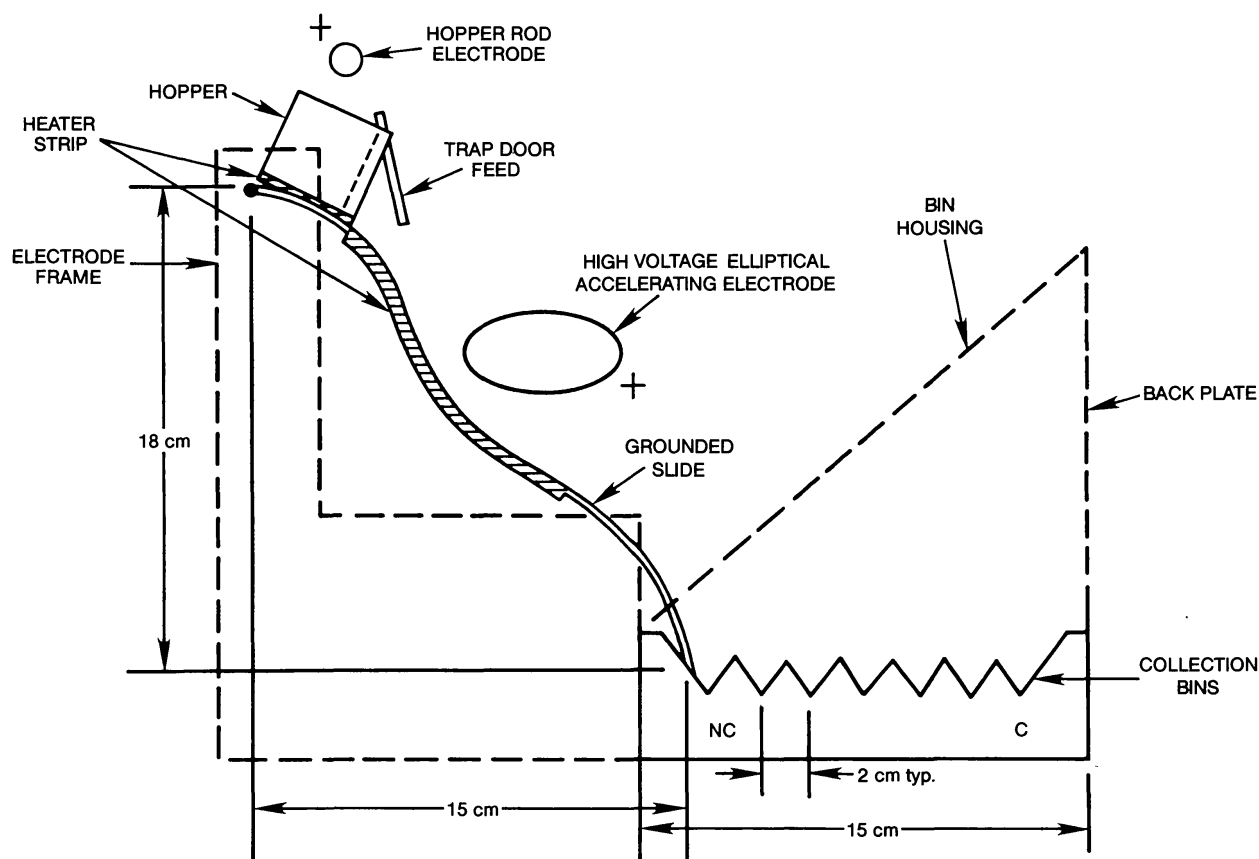


Figure 2. Mineral electrostatic separator: vacuum slide configuration.

For the nitrogen runs, the seven-bin collector was used in a glove box at a positive nitrogen pressure of about one torr. Ilmenite concentration data after one pass were collected in seven runs for four-component simulants in the 0.09–0.15 mm size range. High concentrations of ilmenite reported to bins 2 through 7. Mean grade after one pass was 90 ± 7 wt % (up from 7.9 wt %), and recovery was 67 ± 5 wt % for all runs in nitrogen.

For ilmenite electrostatic concentration in vacuum, the apparatus (Figs. 2 and 3) was scaled to fit a bell jar of approximately 70 liters volume. A trap door hopper was used instead of the vibratory feed to reduce grain bounce and consequent random feed dispersal from the hopper mouth. Grain rebound is especially high in vacuum because both air resistance and sound energy radiation are greatly reduced (Fraas, 1970). A rod-shaped electrode was set above the hopper in an attempt to charge the feed sample by electrostatic induction prior to separation.

After exhausting the bell jar to 10^{-5} torr and heating the sample and slide to approximately 150°C , high voltage (15 kV) was applied to the hopper and the accelerating electrodes in parallel to obtain a field strength of approximately 3 kV/cm in the hopper for 10 minutes before separation. The trap door was then opened to drop the sample onto the slide and into the separating field of approximately 5 kV/cm. Material moved farther toward the back bins than in the air and nitrogen runs, and ilmenite concentrated

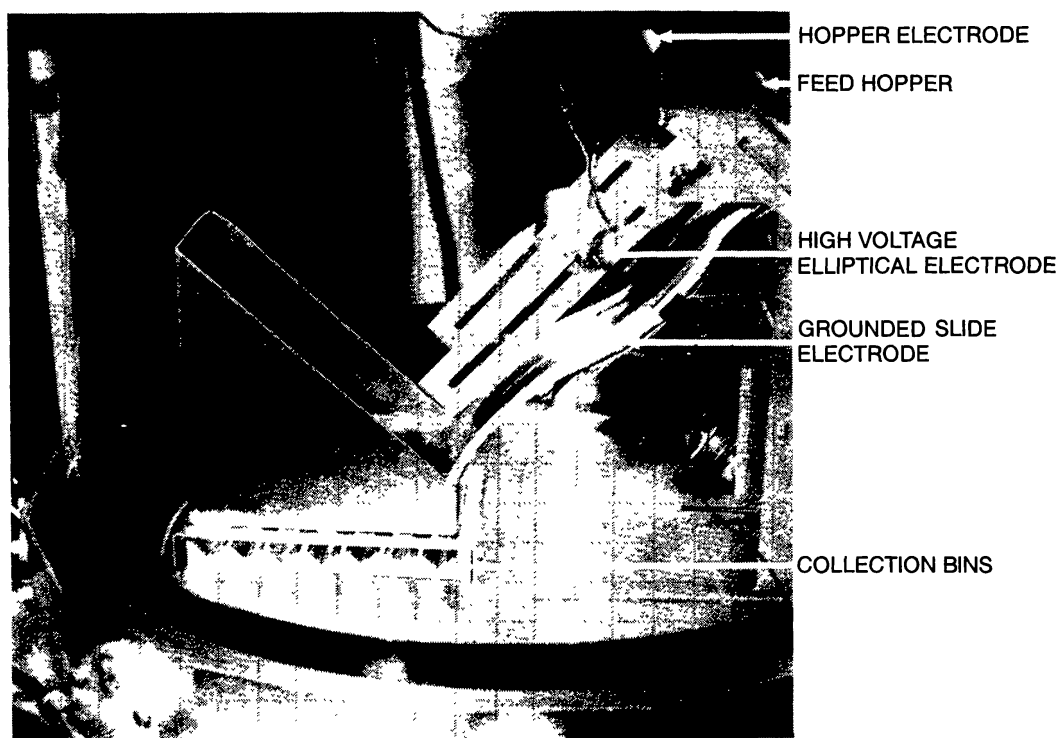


Figure 3. Mineral electrostatic separator slide configuration in vacuum bell jar.

in bins 5, 6, and 7. More material fell outside the bin system as well. Grades of twelve single-pass runs in vacuum were calculated from the cumulative ilmenite concentration in bins 5, 6, and 7. Two recoveries were calculated: R_1 , which excludes material that fell outside the bins, and R_0 , which includes that material. The mean ilmenite grade after one pass of all the vacuum runs was 78 ± 7 wt % (up from 9.8 wt %); $R_1 = 56 \pm 8$ wt % and $R_0 = 49 \pm 8$ wt %.

LUNAR ILMENITE ELECTROSTATIC CONCENTRATION

Table 1 lists lunar ilmenite grade and recovery data of four electrostatic concentration runs on a 0.09–0.15 mm sample of Apollo 11 soil 10084,853. Three of the runs were in dry nitrogen and one in vacuum. The lunar runs in nitrogen were made with the same apparatus and procedure as the simulant nitrogen runs. Similarly, the lunar vacuum run replicated the procedures of the simulant vacuum runs. Lunar ilmenite wt % was also calculated as in the simulant runs.

About 50 vol % of the sample were agglutinates before the first run, and the starting ilmenite content was about 10 wt %. The first nitrogen run in the table and the vacuum run were made without prior removal of agglutinates. However, before the other two nitrogen runs (#2 and #3), the ferromagnetic agglutinates were removed with a permanent magnet of approximately 1500 gauss. The ferromagnetic agglutinates comprised approximately 75 vol % of total sample agglutinates by microscopic count.

Table 1. Lunar Soil Ilmenite Electrostatic Concentration (One Pass)

Apollo 11 Soil Sample 10084, 853 (grain size: 0.09–0.15 mm; sample charging time: 10 min.)							
Run Number	Ambient	Starting Ilmenite Concentration (weight percent)	Sample Feed Temperature (°C)	Maximum Electrostatic Field Strength (kV/cm)	Ilmenite		Conductor Bins
					Grade	Recovery	
					(weight percent)		
1	Dry nitrogen	10	173	4.7	45	24	2–7
2a*	Dry nitrogen	7.3	—	3.0	51	48	2–7
2b†	Dry nitrogen	7.3	—	3.0	66	50	2–7
3a	Dry nitrogen	7.3	193	5.0	51	35	2–7
3b	Dry nitrogen	7.3	193	5.0	62	36	2–7
4a	Vacuum	7.0	154	5.0	29	55	6,7,Tr**
	1.5×10 ⁻⁵ torr						
4b	1.5×10 ⁻⁵ torr	70	154	5.0	37	60	6,7,Tr

* a excludes ilmenite bearing glass and polyphase components.
† b includes ilmenite bearing glass and polyphase components.
** Tr is the catch tray beyond the 7th bin.

Tables 2 and 3 give lunar soil component distributions among the seven collection bins after one pass of electrostatic concentration for the first nitrogen run (#1) and the vacuum run (#4), respectively, with all agglutinates present in both cases. Components microscopically discriminated and confirmed by SEM-EDS analysis were: agglutinate, anorthite, glass, ilmenite, polyphase, and olivine/pyroxene. Olivine and pyroxene counts were lumped together. The polyphase component consisted of lithic fragments that were about half ilmenite, and about half the dark glass grains had ilmenite compositions. The

Table 2. Lunar Soil Ilmenite Electrostatic Concentration in Dry Nitrogen

Soil Component Distribution After One Pass (Run #1, Table 1)										
Site	Weight/ Site (mg)	Aggl	An	Glass	Il	Poly	Px/Ol	Ot	Wt% Il	Wt Il (mg)
(Modal Percent)										
Bin #1	61.2	31	11	11	5	18	22	1	7.3	4.5
2	2.4	11	1	14	24	30	17	3	32	0.8
3	1.1	11	—	9	40	27	14	—	50	0.6
4	0.4	—	—	—	82	14	4	—	87	0.3
5	<0.1	8	—	—	75	17	—	—	81	0.1
6	<0.1	—	—	—	100	—	—	—	100	
7	<0.1	—	—	—	100	—	—	—	100	
S+H+E	11.6	55	9	6	8	10	11	1	11	1.3
Tr	1.0	58	2	3	6	17	13	—	8.7	0.1
Total	77.7									7.7

Aggl—agglutinate; An—anorthite; E—field electrode; H—feed hopper; Il—ilmenite; Ot—other, Two of these grains were metal spherules; Poly—polyphase; Px/Ol—pyroxene/olivine, combined counts; S—grounded slide; Tr—catch tray under bins

Table 3. Lunar Soil Electrostatic Concentration in Vacuum (1.5×10^{-5} torr)

Soil Component Distribution After One Pass (Run #4, Table 1)									
Site	Weight/ Site (mg)	Aggl	An	Glass	Il	Poly	Px/Ol	Wt% Il	Wt Il (mg)
(Modal Percent)									
Bin #1	16.6	75	4	3	3	6	8	4.4	0.7
2	26.8	71	4	3	2	2	17	3.0	0.8
3	8.0	60	4	4	2	4	26	3.0	0.2
4	3.3	56	6	4	5	3	26	7.3	0.2
5	1.8	58	7	3	10	6	16	14.3	0.3
6	1.9	45	4	3	18	13	17	25.0	0.5
7	3.5	35	4	2	20	15	25	27.0	0.9
S+H+E	0.4	46	26	1	3	1	19	4.4	–
Tr	3.6	33	5	1	25	7	29	33.0	1.2
Total	65.9								4.8

Aggl—agglutinate; An—anorthite; E—field electrode; H—feed hopper; Il—ilmenite; Poly—polyphase; Px/Ol—pyroxene/olivine, combined counts; S—grounded slide; Tr—catch tray under bins

discrepancy in glass, polyphase, and agglutinate counts between Tables 2 and 3 resulted from a confusion among those components when counting run #1.

In the first nitrogen run, with all the agglutinates present, approximately 12 wt % of the sample coated the field electrode and about 3 wt % clung to the slide and hopper that together comprise the electrical ground. By contrast, in a vacuum run that also contained all the agglutinates, only 0.5 wt % of the sample clung to the slide, hopper, and field electrode combined. In both cases, half this material was agglutinates. Nitrogen ion and electron charging of the agglutinate-rich sample of the first run probably account for the differences.

Lunar ilmenite behaved like a semi-conductor in all the runs. Although it reached 100% concentration in bins 6 and 7 of the first nitrogen run (Table 2) and 80 and 90 wt % in the other nitrogen runs, only a few grains (<1 wt % of the sample) appeared there. In the vacuum run, by contrast, about 14 wt % of the sample reported to bins 6 & 7 and beyond (into the catch tray) but only at a combined ilmenite grade of 29 wt % (up from 7 wt %). Recovery for the vacuum run was 55 wt % (Tables 1 and 3). Mean ilmenite grades and recoveries were not calculated for the lunar sample runs because of the small number of runs and their differing conditions. However, the identical grade (51 wt %) for runs #2a and #3a under the same conditions suggests reproducibility. Adjusted grades and recoveries that include half the glass and polyphase fractions in the conductor bins to reflect their ilmenite contributions are also reported for runs 2, 3, and 4 in Table 1. After adjustment, the grade of the vacuum run increases from 29–37 wt %, and runs #2 and #3 in nitrogen increase from 51 wt % to the mid 60s. The calculation was not done for run #1 because of uncertainty in the glass and polyphase counts.

Agglutinates behaved like non-conductors in all the lunar runs regardless of whether or not the ferromagnetic agglutinates were removed. Over 90% of sample agglutinates

reported to the non-conductor bin or bins and the electrodes combined, and about 5% were spread over the remaining bins (Tables 2 and 3).

DISCUSSION

Significant one-pass concentrations of ilmenite were obtained in all the runs from the average eleven-fold increase for the simulant runs in nitrogen (7.9–90 wt %) to a four-fold increase in the lunar soil run in vacuum (7.0–29 wt %). Grades in nitrogen and air were significantly better than in vacuum probably for the following reasons:

1. Gas ionization products contributed to feed charging in air and nitrogen.
2. The absence of vibratory sample feed in vacuum may have reduced the efficiency of mineral contact and induction charging compared with the atmospheric runs.
3. Partial density segregation of ilmenite occurred as the feed fell in gas at atmospheric pressure.
4. Feed transit time in the separating field was greater in air and nitrogen than in vacuum because of air resistance.
5. The ilmenite concentration in the seventh bin of the vacuum runs was reduced due to two effects.

a. The horizontal component of acceleration imparted to feed grains by the slide (see Fig. 4) transported a few percent of the feed to the far bins in vacuum at zero electric field and led to a dilution of ilmenite separates reporting to the conductor bins with the field on. By contrast, none of the feed was transported mechanically to the far bins in air or in nitrogen at zero field.

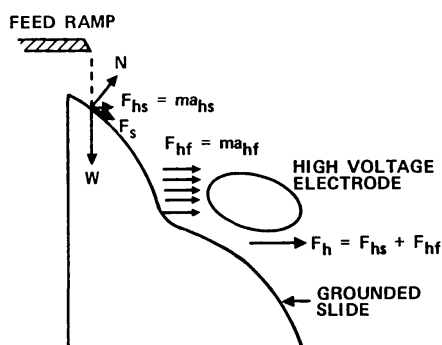


Figure 4. Force diagram of mineral grain on slide.

LEGEND

- a_{hf} — HORIZONTAL COMPONENT OF GRAIN ACCELERATION DUE TO ELECTRIC FIELD
- a_{hs} — HORIZONTAL COMPONENT OF GRAIN ACCELERATION DUE TO SLIDE
- F_h — HORIZONTAL FORCE ON GRAIN
- F_{hf} — HORIZONTAL FORCE ON GRAIN DUE TO ELECTRIC FIELD
- F_{hs} — HORIZONTAL FORCE ON GRAIN DUE TO SLIDE
- F_s — FORCE ON GRAIN PARALLEL TO SLIDE
- m — MASS OF GRAIN
- N — FORCE ON GRAIN PERPENDICULAR TO SLIDE
- W — WEIGHT OF GRAIN

b. A back plate behind the seventh bin, for containment of material within the bin system (see Fig. 2), tended to deflect material striking it toward the seventh bin. As a consequence, more material was collected in the seventh than the fifth and sixth bins in the vacuum runs. That also may have contributed to dilution of the ilmenite concentrate and suggests that a more extended bin system might result in better grades on the slide apparatus in vacuum.

Recovery in the vacuum lunar soil run was higher than in the lunar sample runs in nitrogen and is in part due to the larger sample fraction that reached the conductor bins in vacuum. In the simulant runs, higher recoveries also tended to be associated with lower grades. Mean bin recovery (R_1) of the simulant runs in vacuum lies between the recoveries in air and nitrogen and probably reflects performance variability due to grain bounce within and outside the bin system in vacuum.

CONCLUSIONS

Significant concentrations of lunar soil ilmenite from 7.3–51 wt % have been obtained in one pass of an Apollo 11 soil sample through a slide-type electrostatic separator in nitrogen after the ferromagnetic agglutinates had been removed with a permanent magnet. Ilmenite was concentrated in the same sample and in a similar apparatus (Figs. 2 and 4) from 7.0–29 wt % after one pass in vacuum with all the agglutinates present. Lunar soil ilmenite recovery was 55 wt % in the vacuum run and lower in nitrogen (ranging from 24–48 wt % in three separate runs).

It should be emphasized that the lower limit of the grain sizes tested in this study was 0.09 mm. Mineral electrostatic separation of fines in vacuum has not yet been investigated, presumably because there has been no terrestrial commercial application. However, Carta *et al.* (1964) reported improved one-pass grades and recoveries of a graphite ore (7.7% carbon) concentrated in air at reduced pressure (120 torr) compared with concentration at one atmosphere in two commercial drum electrostatic separators. Grades and recoveries were doubled in some cases even for grain size ranges less than 0.075 mm. Carta *et al.* attributed the improvement to several factors: (1) enhanced ionization charging of the feed due to enhanced corona current at the lower gas pressure, (2) improved contact of feed and grounded electrode (the drum), (3) increased uniformity of grain trajectories in all size ranges, and (4) reduced agglomeration of fines and dusting of the coarser grains with fines. Factors 2 through 4 are likely to be even more enhanced in vacuum. For that reason, mineral electrostatic separation of fines in vacuum is an important follow-up investigation to this study, especially for lunar field applications.

Electrostatic separators offer the advantages of low power consumption and mass and efficient high voltage generation in a vacuum environment such as the lunar surface. However, existing separator designs are intended for atmospheric operation and take advantage, either by design or by accident, of atmospheric effects that enhance separation performance for grain sizes >0.1 mm such as (1) mineral feed charging by air ionization in the separating field, (2) partial density segregation of mineral grains falling through air in the feed path of the separator, or (3) increased feed transit time in the separating

field due to air resistance. Existing designs with minor modifications would probably work very well in a gas environment established on the Moon, but major modifications are required for efficient vacuum operation.

Multistage electrostatic separation systems like those used in all commercial electrostatic mineral concentrators should raise grades and recoveries to 90%+ levels (e.g., Kelly and Spottiswood, 1982), provided the soil ilmenite is sufficiently liberated. Even without additional liberation, lunar soil ilmenite grades in the size range tested could reach the high 70s because combined glass and polyphase soil components that reported along with ilmenite to the conductor bins and beyond were comparable to ilmenite in abundance (Table 3) and are about half ilmenite in composition. Agglutinates are the major component of ilmenite bearing mare soils (Papike *et al.*, 1982), and their divergent electrical behavior to ilmenite is an indication that the two components are separable electrostatically. This work suggests that the best sequence for concentrating lunar soil ilmenite would be magnetic extraction of the ferromagnetic agglutinates followed by electrostatic concentration of ilmenite in the non-ferromagnetic soil fraction.

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