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APOLLO 11 ILMENITE REVISITED

WCSAR-TR-AR3-9201-3

Technical Report

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January 1992

Prepared for Space 92, The Third International Conference on Engineering, Construction, and Operations in Space, 31 May–4 June 1992, Denver CO

Apollo 11 Ilmenite Revisited

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Abstract

Various investigations have shown (1) that lunar regoliths are extremely complex materials, (2) that ilmenite is a minor component (8 to 10%) of high-Ti regoliths, and (3) that most of the ilmenite is interlocked with other minerals or glass in regolith particles. A review of polished sections of Apollo regolith samples that I studied in 1970 in general confirms these findings.

Beneficiation of regolith will not be easy; there is no certainty that a high-grade ilmenite concentrate can be produced. Magnetic and electrostatic separation have promise for removing much of the roughly one-third of the regolith particles that contain little or no ilmenite. However, further beneficiation by these methods can only be effective if confined to the finest size fractions. Sizing of regolith to remove coarser fractions will therefore be of paramount importance.

The problems of large-scale beneficiation need to be addressed, beginning with tests on kilogram-size samples of actual regolith. In view of the critical importance of lunar oxygen production to lunar development, acquisition of large samples of high-Ti regolith should be a top priority for the next lunar mission.

Introduction

A critical problem in planning further exploration of the Moon and development of lunar resources is the provision of the oxygen and water essential for life support. It is clear that it will not be feasible to supply these substances from Earth, and their procurement from asteroids, if it ever proves possible,

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is too far in the future to be considered in present planning for lunar exploration. Both oxygen and water must therefore be obtained from resources on the Moon.

Hydrogen for making water is not a problem. The gas is present in very large amounts in the regoliths of certain lunar maria and can be extracted simply by heating regolith. Oxygen is present in essentially unlimited amounts in the silicates and oxides of lunar rocks and regoliths, but its extraction is much more difficult than hydrogen extraction. Various processes of oxygen extraction have been under investigation for a number of years (Taylor and Carrier, in press). Of these, reaction of ilmenite with H_2, C, CO, CH_4, or CO-Cl_2 plasma has received much attention, especially the treatment of regolith ilmenite with hydrogen. When hydrogen is used, the reactions involved are

(1) $FeTiO_3$ (ilmenite) + $H_2 \rightarrow Fe + TiO_2 + H_2O$

(2) $2\ H_2O \rightarrow 2\ H_2 + O_2$, by electrolysis.

The hydrogen produced in the second reaction can obviously be recycled.

Ilmenite in the Regolith of the Apollo 11 Landing Site

Abundance of Ilmenite. The Apollo 11 mission landed in the southwestern part of Mare Tranquillitatis. Ilmenite is abundant both in the high-Ti basalts of the mare and in the regolith derived from them. Modal analyses of Apollo 11 rocks show ilmenite contents ranging from 10.1 to 24% by volume (Agrell et al., 1970; Bailey et al., 1970; Brown et al., 1970; Cameron, 1970; Carter et al., 1970; Chao et al., 1970; Dence et al., 1970; French et al., 1970). Modal analyses of bulk samples of regolith are not available. Bulk chemical analyses of Apollo 11 regolith fines show TiO_2 contents of 7.4 to 8.5%, which would indicate ilmenite contents of about 14 to 16 wt. % if all the TiO_2 were present in ilmenite. However, Fredericksson and others (1970) found that part of the TiO_2 is present in glass spherules and fragments which have up to 12 wt. % TiO_2 but mostly contain 3 to 8 wt. % TiO_2. The same authors reported that scoriaceous glass forming a large part of the glassy fraction of the lunar fines contains 7.29% TiO_2, almost the same as the TiO_2 contents of bulk samples of the fines. Modal analyses of regolith in the literature report only percentages of free ilmenite; amounts of ilmenite contained in rock particles are not given.

To provide additional information on the ilmenite content of Apollo 11 fines, point counts have been made of ilmenite in polished sections of fractions of two samples of the fines. The results are given in Table 1. The table indicates that the 105-350 μm fractions of 10084 and 10085 are essentially identical in

Table 1

Modal Percent Ilmenite From Point Counts

	(1)		(2)		(3)		(4)	
	Points	%	Points	%	Points	%	Points	%
Ilmenite	98	9.8	97	9.7	83	9.1	75	7.5
Other minerals plus glass	902	90.2	903	90.3	827	90.9	925	92.5
Total points	1000		1000		910		1000	

(1) 10084/1418, 105-350 μm fraction.
(2) 10085/673, 105-350 μm fraction.
(3) 10084/1020, 105-350 μm fraction.
(4) 10084/1021, 350-1000 μm fraction.

total ilmenite content. The ilmenite content of the 350-1000 μm fraction of 10084 appears to be definitely lower. It would be desirable, however, to have counts on additional portions of the 350-1000 μm fraction.

Crystal Habits of Ilmenite

As various investigators have noted, ilmenite in Apollo 11 basalts and basaltic rock fragments in regolith shows a rather wide range of crystal habits; some common habits are displayed in Figure 1. In the prevailing holocrystalline rocks, tabular to thinly platy crystals are characteristic, but in a few rocks ilmenite is irregularly intergrown with silicates. In the finest-grained rocks and especially in the hyalocrystalline rocks, a spectacular array of plumose and skeletal forms is found. However, a count of grains in a polished thin section of the 105-350 μm fraction of 10084 showed that only about 8% of the ilmenite-bearing rock particles contain plumose and skeletal forms of the mineral.

Grain Size of Ilmenite

Out of 16 crystalline rocks brought back by the Apollo 11 mission (Schmitt and others, 1970), 3 are very fine-grained ($<$ 0.1 mm average diameter), 6 are fine-grained (0.1-0.5 mm), 3 are medium-grained (0.5-1.0 mm), and 4 are coarse-grained (1.0-5.0 mm). The samples were undoubtedly taken to represent the range of rock types observed by the astronauts. They should not be taken to indicate the proportions of rocks of the various grain-size classes in the regolith. The polished samples prepared from fractions of the regolith should give a much more accurate picture of grain size variation in

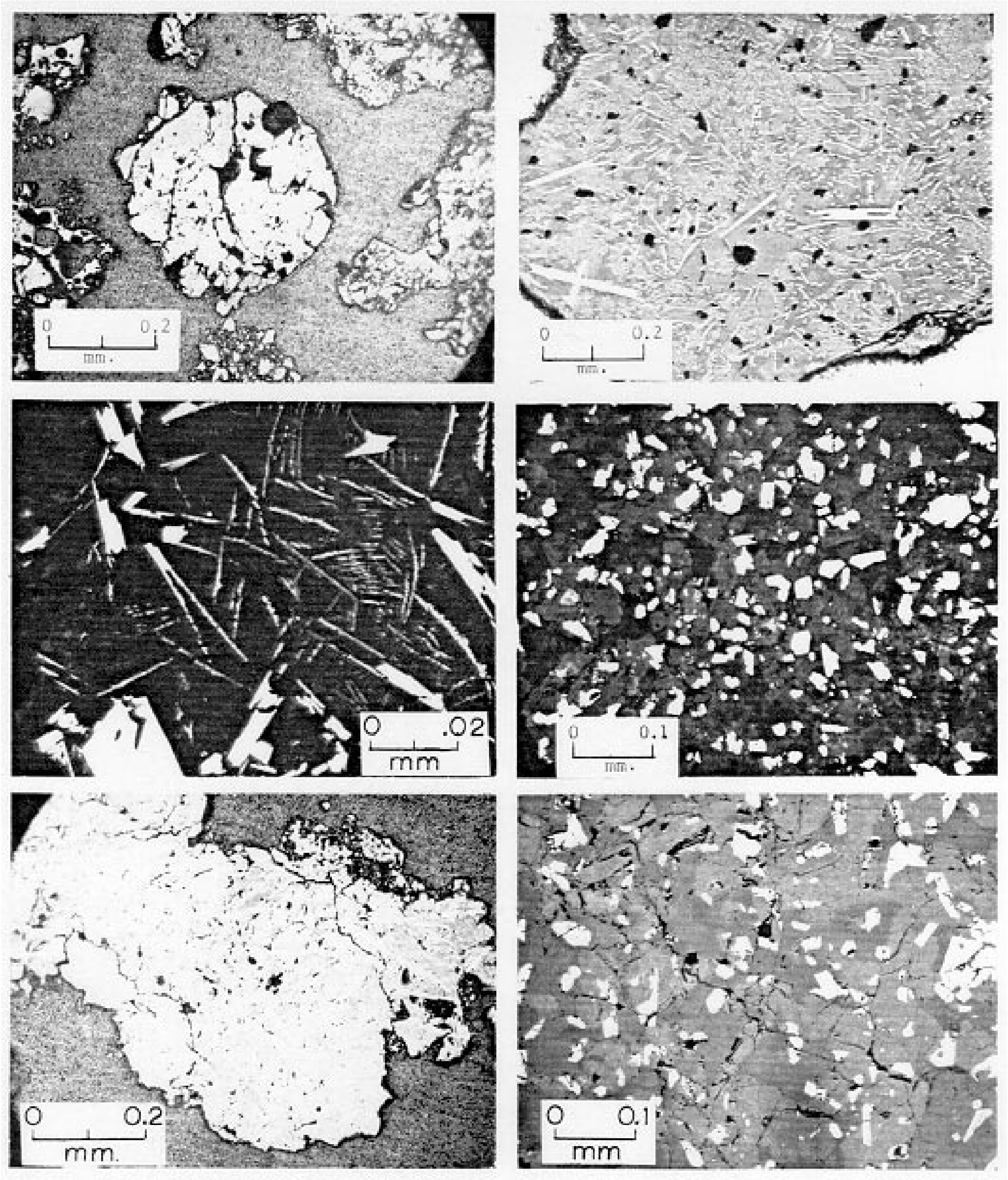

Figure 1. Some common forms of ilmenite in regolith particles as seen in reflected light. Ilmenite crystals are white; silicate minerals are gray or black. The two center photographs were taken in oil immersion. A few crystals of armalcolite, slightly darker than ilmenite, are present in the left center photograph.

Table 2

Regolith Particles in the 105-350 μm Fraction of Regolith Fines, Grouped According to Maximum Diameter of Contained Ilmenite Crystals

	(1)		(2)		(3)	
Ilmenite - Max. Diam.	No. of Particles	%	No. of Particles	%	No. of Particles	%
All > 50 μm	49	4.8	50	4.5	104	9.3
Most > 50 μm	48	4.8	50	4.5	73	6.5
Most < 50 μm	57	5.6	46	4.2	57	5.1
All < 50 μm	503	49.1	562	51.0	463	41.3
No ilmenite	368	35.9	392	35.6	425	37.8
Totals	1025	100.2	1100	99.6	1122	100.0

(1) 10084, 1418
(2) 10084, 1020
(3) 10085, 673

the rocks from which the regolith is derived. Two polished sections of material from the 105-350 μm fraction of sample 10084 and one polished thin section of the same fraction of sample 10085 were therefore studied. Each section was traversed along parallel lines 0.4 mm apart, and with the aid of a micrometer ocular each ilmenite-bearing regolith particle intersected was classified into one of four groups according to the maximum diameter of its ilmenite crystals. Results are shown in Table 2. It is evident that most of the ilmenite is very fine-grained. Very little of the ilmenite in the 105-350 μm fraction has been unlocked from silicates. Free ilmenite grains in the three sections prepared from the 105-350 μm fractions number only 9, 18, and 10, respectively. Furthermore, it is clear from inspection of the polished sections and from grain size measurements that even in the 20-50 μm fraction of the regolith, much of the ilmenite will be locked with silicates or glass.

Table 3 shows results of examination of polished sections prepared from the +350 μm fractions of the fines. The number of particles available for counting is limited, but again, most of the ilmenite in particles containing the mineral is less than 50 μm in maximum diameter. There are no free ilmenite particles in the sections.

It is important to note that the grains classified as ilmenite-bearing for purposes of Tables 2 and 3 show a wide range of ilmenite content, from

Table 3

Regolith Particles in the +350 μm Fraction of Regolith Fines, Grouped According to Maximum Diameter of Ilmenite Crystals

Ilmenite Crystals Maximum Diameter	(1)	(2)	(3)	(4)	Summary No.	%
All > 50 μm	23	0	1	8	32	8.4
Most > 50 μm	27	3	4	28	62	16.2
Most < 50 μm	28	7	15	10	60	15.7
All < 50 μm	114	14	6	2	136	35.6
No ilmenite	88	2	0	2	92	24.1
Totals	280	26	26	50	382	100.0

(1) 10084/1021
(2) 10085/672
(3) 10085/524
(4) 10085/522

more than 20% to only traces of the mineral. As indicated by the tables, roughly one-third of the particles in the polished sections show no ilmenite at all. Removal of these particles from the feed to the hydrogen reduction plant should be a major objective of beneficiation.

Degree of Exposure of Ilmenite

The polished sections show that most of the ilmenite crystals in the 105-350 μm fractions of regolith samples are not exposed on the surfaces of regolith particles. The same will certainly be true for the 50-105 μm fraction, but in the -50 μm fraction both degree of exposure on particle surfaces and number of free ilmenite particles should be considerably enhanced. The relation should be an exponential increase in surface area of exposed ilmenite with decrease in grain size.

Implications for Beneficiation

For the hydrogen reduction process it is desirable to have a feed with the highest possible content of ilmenite. Criswell and Waldron (1982) suggested concentration by electrostatic separation. Gibson and Knudsen (1986) suggested a concentrate with 80-90% ilmenite, to be produced by magnetic and electrostatic separation. Other discussions of the reduction process (e.g., Gibson et al., 1990) simply assume that such a concentrate will be available.

Gibson and Knudsen (1990) have calculated mining and processing parameters for a plant of 150 tonnes of lunar LOX, using hydrogen reduction. The calculations are based on a feed of pure ilmenite to be obtained by beneficiation of regolith averaging 5% ilmenite. However, the data presented here indicate that producing a concentrate of high ilmenite content from the regolith fines will be very difficult. The first requisite for producing concentrates by physical means on Earth is breaking down mineral aggregates so as to free a desired mineral or minerals from associated unwanted minerals (the gangue). The Apollo 11 regolith is already a crushed material, but unlocked (free) ilmenite will be found in any abundance only in the finest fractions. Even in the -50 μm fraction, much of the ilmenite will occur not as free grains but as a component of rock particles. Free grains in abundance will be found only in the -10 μm fraction forming about 16% of the soil (Criswell and Waldron, 1982). In the 10-20 μm fraction, an additional 11% of the fines, there should be some free ilmenite, but more will occur as a component of aggregates. Concentrating a mineral from such fine and texturally diverse material is difficult enough on Earth and will be more difficult on the Moon, where the range of applicable techniques will be much more limited.

The consequences of the size, crystal habits, and modes of occurrence of ilmenite in the Apollo 11 regolith are indicated in the results of magnetic separation performed by Taylor and Oder (1990). Each of a series of size fractions of 10084 fines was separated into low, intermediate and high magnetic susceptibility fractions. The results show that plagioclase tends to concentrate in low susceptibility fractions, ilmenite in intermediate fractions, and agglutinates and melt rock in high susceptibility fractions. However, no clean separations of regolith components were obtained even from the finest size fraction treated (20-44 μm). The ilmenite fractions of various intermediate magnetic fractions are reported as ranging from 12 to 16%, based on particle counts.

Apollo 11 regolith is mature. Concentrations of ilmenite in intermediate magnetic fractions obtained from an Apollo 17 sample of immature regolith are reported to range up to 29%. However, particles counted as “ilmenite” in the fractions of regolith samples are stated to be those containing 40% or more ilmenite, so that actual ilmenite percentages in the concentrates must be less than those reported.

The difficulty of concentrating ilmenite is what would be predicted from Tables 2 and 3 and study of the polished sections of regolith fines. However, it appears that magnetic beneficiation can be used to get rid of a substantial percentage of the particles that contain little or no ilmenite, since these will pass into the low and high susceptibility fractions.

Electrostatic beneficiation has been under consideration as a means of concentrating ilmenite, alone or in combination with magnetic separation (e.g., Criswell and Waldron, 1982; DeLa'O et al., 1990). However, the results of electrostatic separation are likely to be similar to those of magnetic separation, because the same characteristics of ilmenite occurrence in regolith that impede magnetic concentration will also be obstacles to electrostatic separation. Yet electrostatic separation should aid in removing particles containing little or no ilmenite, and a combination of the two processes of beneficiation might produce concentrates somewhat higher in grade than those produced by either method alone.

Gravity methods are widely used on Earth for separation of ilmenite from associated minerals. The silicate and glass components of mare regoliths have specific gravities ranging from about 2.7 to about 3.5, whereas ilmenite has a specific gravity of about 4.75. Again, however, since ilmenite is mostly locked with other regolith components, no clean separation will be possible. Even if only the finer size fractions of regolith are treated, a high proportion of middlings is likely. An additional problem is that gravity methods operate best in fluid media in a high gravity field.

It may well be that sizing and rejection of $+50$ μm fines will prove to be the most effective means of beneficiation of regolith. The -50 μm fraction will have a higher proportion of free ilmenite than the rejected fraction. Furthermore, hydrogen reduction is not dependent solely on the amount of free ilmenite. Ilmenite exposed at the surfaces of polyphase particles will also be available for reaction with hydrogen. As noted earlier, the proportion of such ilmenite will certainly be much higher in the finer fractions. Sizing will therefore have a doubly beneficial effect on availability of ilmenite for reaction with hydrogen. In addition, development of sizing methods is important to the recovery of helium-3, since most of the helium in regolith is in the -100 μm fraction (Cameron, 1990). Development of effective means of sizing regolith therefore deserves vigorous investigation.

Summary and Conclusions

The lunar regolith at the Apollo 11 site, and probably the regolith of Mare Tranquillitatis in general, is a very complex material, with a considerable range of textural types of basalts. Much of the ilmenite is very fine-grained and even in the very fine size fractions of the regolith is intergrown with silicates or rock glass, or with both. Magnetic and electrostatic separation, combined with sizing to reject all but the finest fractions of the regolith, appear to offer the most promise for beneficiation, but production of high-grade ilmenite concentrates on the scale necessary for adequate oxygen production on the Moon will probably not be achievable. Estimates of mining and processing capacity and equipment based on availability of a high-ilmenite feed from regolith therefore appear unrealistic.

The writer's findings are similar to those of Heiken and Vaniman (1990), who therefore suggest producing ilmenite directly from high-titanium basalt, either bedrock or boulders associated with impact craters. However, use of this source would present its own problems, among them the necessity for massive equipment for crushing and grinding (cf. DeLa'O et al., 1990). It would seem desirable first to determine just what degree of concentration of ilmenite can actually be achieved by beneficiation of regolith and then assess the practicability of using it as a source of ilmenite for hydrogen reduction.

The feasibility of producing a usable ilmenite concentrate can only be established by testing beneficiation methods on much larger samples of regolith than are presently available. Beneficiation of gram-size samples in the laboratory is one thing; beneficiation of large tonnages of material on the Moon will present quite different problems. Procurement of kilogram-size samples should be a major objective of future missions to the Moon.

Acknowledgements

This work was supported by the Wisconsin Center for Space Automation and Robotics (WCSAR), by NASA, Office of Commercial Programs, under Grant NAG-W975.

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