Beneficiation of Ilmenite from Lunar Analog

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Abstract

The results reported here were obtained on a meteoric eucrite sample called "Millbillillie Sample #173. Optical microscopy studies of the sample showed it to consist of 4 volume percent ilmenite, 2 volume percent troilite, and 94 percent transparent gangue. The transparent gangue consisted of 46 percent feldspar (anorthite), 38 volume percent pyroxenes, 10 volume percent olivines, and 6 percent opaques. Troilite was present in minor quantities. The head sample analyzed 15.5 percent total iron, 11.8 percent ferrous iron, and 0.37 percent titanium. Screen assay analyses of the 30, 100, 200, and 400 US mesh screen fractions showed that minor concentration of titanium occurred in the 200 x 400 and -400 mesh screen fractions.

Scanning electron microscopy (SEM) studies of the bulk sample showed the presence of a variety of ilmenite grains, ranging from 50 micrometers (μm), down to less than 1 μm without any evidence of liberation. Electron Diffraction scans (EDS) confirmed the ratio of Fe to Ti in the ilmenite grains.

Dry magnetic separation in a Frantz Isodynamic Separator was found to be effective only at sizes finer than 150 μm (100 US mesh) and more so at 200 mesh (74μm). In each case, dedusting of the sample to remove -400 mesh (-0.037 μm) fines was required. Liberation size was determined to be 200 mesh and finer.

The highest grade concentrate assaying 3.45 percent Ti was produced by magnetic separation of the -200 + 400 mesh screen fraction assaying 0.44 Ti (from a -30 mesh sample) at a current setting of 0.35 AMP. This concentrate contained 21.2 percent of the Ti values in the screen fraction with 2.72 weight percent of feed to test.

The results can be projected to a sample stage ground to -200 mesh. Magnetic separation of the -200 + 400 mesh (-0.074 + 0.037 μm) should produce a concentrate accounting for 1.41 weight percent of the feed. This concentrate will analyze 3.45 percent Ti and contain 10.3 percent of the Ti values in the feed. By changing the Frantz Magnetic Separator settings, a lower grade concentrate analyzing 0.98 percent Ti can be produced at an increased recovery of 25.4 percent. The concentrate weight will be 11.7 percent of the feed. It must be emphasized that improved grades and recoveries can be obtained with the -400 mesh fines. However, beneficiation of these extremely fine materials is not possible in a practical process scheme.

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Several dry beneficiation processes were tested on -10 mesh and -30 mesh samples. These included the following:

- o Vertical Electrostatic (ES) Free Fall Separator (V-STAT)
- o Electrostatic (ES) Plate Separator
- o Rare Earth Drum Magnet Separator (KHD)
- o High Tension (HT) ES Roll Separator
- o High Intensity Lift Roll Magnet
- o Magstream Gravity Concentrator

The most significant physical upgrading was achieved in a Two-pass High Tension Electrostatic Roll Separator. The feed to the test consisted of the 200 x 400 mesh fraction of the -30 mesh sample. The 200 x 400 mesh fraction constituted 12 percent of the sample assayed 0.42 percent Ti, and contained 13.3 percent of the Ti values. The conductor material from the first pass was retreated in a second stage to produce a concentrate analyzing 2.73 percent Ti, with 39 percent Ti recovery. The two-pass conductor retreat product accounted for 5.44 weight of feed to the test, or 0.67 percent of the original -30 mesh sample. It is most probable that a single pass ES separation was just as effective as the two-pass ES test, since the conductor weight percentages for the single pass and two-pass tests were comparable.

By projecting the above results to a -200 mesh grind, a two-pass ES conductor retreat product accounting for 2.88 weight percent of the total feed should be possible. This product is expected to analyze 2.73 percent Ti, with 18.9 percent recovery of the Ti values in the total feed sample.

The free-fall ES Separator appeared to be effective in removing fines from the coarser sizes in the sample. This technique could be useful in dedusting lunar samples.

Wet Gravimetric-magnetic separation in the Magstream apparatus was successful in upgrading the -200 + 400 mesh fraction (at an apparent specific gravity of 4.25) from 0.42 percent Ti to 1.06 percent Ti, with 52.6 percent recovery in a concentrate representing 20.7 weight percent of feed to the test. This included the paramagnetic product, which was 6 weight percent, and contained 19.3 percent of the Ti values, at an assay of 1.37 percent Ti.

The above results can be projected to a -200 mesh grind sample, containing 51.9 percent as -200 + 400 mesh, and 48 percent of the Ti values. Magstream Separation would produce a heavy concentrate with 10.74 weight percent and 27.3 percent recovery at a grade of 1.06 percent Ti. It would be interesting to determine if ES roll separation of this heavy fraction would improve grade with minimum recovery losses.

Future work along the following lines is recommended on the Eucrite Sample:

- o Wet Magstream, followed by dry ES roll separation of the heavy fraction.
- o Dry Magnetic separation, followed by dry ES roll separation.
- o Test V-Stat separator for efficiency of dedusting.
- o Complete SEM and EDS scans of concentrates produced from tests with the ES two-pass separator, and the Frantz Magnetic Separator.

The additional data to be generated on the eucrite will determine the scope of process, engineering, and scientific studies on the very limited quantities of lunar material (5 to 10 grams) to be made available in 1993.

Introduction

The results summarized in this report are a continuation of beneficiation test work reported previously in *Progress Report Number 1* on Millbillillie Sample #173, characterized as an eucrite in the literature. This report summarizes details of Magnetic, Electrostatic, and Magstream testing completed on the Millbillillie sample.

Sample

The mineralogical makeup of the Millbillillie sample was previously discussed in extensive detail in *Progress Report Number 1*, and will not be repeated here. Sample preparation procedures can also be found in the reference cited earlier.

The screen-size distribution of the -10 mesh, -30 mesh, and -200 mesh samples used in the Electrostatic, Magstream, and Magnetic Separation testing is given in Tables 1 and 2. It is clear that the titanium analysis is quite low at 0.36 percent Ti to 0.39 percent Ti. The analyses for total iron, ferrous iron, and titanium are in the range of 13.73 to 15.08 percent, 12.78 to 13.09 percent, and 0.36 to 0.39 percent, respectively. Previous SEM studies showed the presence of a variety of liberated ilmenite grains ranging from 10 micrometers (10 μm) down to less than 1 μm, as well as 1 μm unliberated and liberated grains. Electron diffraction scans (EDS) confirmed the ratio of Fe to Ti in the ilmenite grains.

TABLE I

Screen Assay Distribution of Samples used in Magstream Separation Tests

US mesh size	Direct Assays (%) Wt. %	Tot. Fe	Fe^{++}	Ti	Percent Distribution Tot. Fe	Fe^{++}	Ti
-30 + 100	34.4	16.2	13.8	0.34	38.7	37.8	30.8
-100 + 200	20.2	16.6	14.6	0.35	23.3	23.4	17.9
-200 + 400	17.9	14.4	12.4	0.43	17.9	17.6	20.5
-400	27.5	10.5	9.7	0.44	20.1	21.2	30.8
Calc'd Head	100.0	14.4	12.6	0.39	100.0	100.0	100.0

TABLE 2

Head Assays and Screen Size Distribution for Samples
Used in Electrostatic and Magnetic Separation Tests

Millbillillie Sample (-30 Mesh Grind)

Head Assay 14.2% Tot. Fe, 12.9% Fe^{++}, 0.36% Ti

U. S. Mesh Size	Direct Wt % Ret.	Assays (%) Tot. Fe	Fe^{++}	Ti	Percent Distribution Tot. Fe	Fe^{++}	Ti
-30 +200	72.74	14.30	13.50	0.37	75.8	76.8	69.4
200 x 400	11.97	13.31	12.39	0.42	12.6	11.6	13.3
-400	15.29	10.50	9.65	0.44	11.7	11.5	17.3
Calcd Head	100.00	13.73	12.78	0.39	100.0	100.0	100.0

Millbillillie Sample (-200 Mesh Grind)

Assay Head 14.4 % Tot. Fe 12.4% Fe^{++} 0.43% Ti

Head Assay Not Available

-200 + 400	51.85	17.20	15.50	0.34	59.1	61.4	48.4
-400	48.15	12.80	10.50	0.39	40.9	38.6	51.6
Calcd Head	100.0	15.08	13.09	0.36	100.0	100.0	100.0

Millbillillie Sample (10 mesh Head Sample)
(after reducing -10 mesh +200 mesh to -200 mesh and screen)

-200 + 400	33.20	15.50	14.58	0.37	7.2	6.9	32.4
-400	56.70	13.95	12.90	0.37	56.2	56.2	56.8
-400	10.10	10.00	8.95	0.43	36.6	36.9	10.8
Calc'd Head	100.00	14.07	13.02	0.37	100.0	100.0	100.0

Magnetic Separation

Samples

The analyses and screen size distribution for the samples tested were described earlier.

Equipment, Procedures, and Results

Dry Magnetic separation was completed on campus in the Chemical Engineering Department, using the Frantz Isodynamic Separator at a series of current, forward and side slope settings. Ferromagnetic particles were removed by a head magnet before the separation. The data from these tests are summarized in Table 3. The concentrate produced at 0.35 AMP is identified as the final product. The data on the combined 0.35 x 1.25 AMP product is also given for comparison. The head assays for all the screen fractions tested are also noted. Individual test data are compiled in Appendix 2, and the analytical results in Appendix 3.

Discussion of Results

An analysis of the data will lead to the following conclusions:

1. Dry magnetic separation is effective only at sizes finer than 100 mesh (150 μm). In each case, dedusting of the sample to remove -400 mesh (37 μm) fines is essential.
2. The sample has to be ground to -200 (74μm) mesh for magnetic separation to be effective.
3. The highest grade concentrate assaying 3.45 percent Ti was produced by magnetic separation of the -200 + 400 mesh screen fraction at a current setting of 0.35 AMP. This concentrate contained 21.2 percent of the titanium values in the screen fraction and accounted for 2.72 weight percent of the sample used in the test.

By changing vibrator feed rate, side slope, and forward slope settings, the Ti recovery can be increased to 52.4 percent, but the grade decreases to 0.98 percent Ti. The concentrate weight percent was 22.6 of the sample used in the testing.

The test results described above on the -200 + 400 mesh fraction of -30 mesh sample can be used to project grades and recoveries of the sample stage ground to -200 mesh as discussed below. By stage grinding, a -200 mesh sample can be produced that contains 48.4 percent of the titanium at a grade of 0.34 percent, as shown in Table 2.

Magnetic separation of the -200 + 400 mesh screen fraction of -200 mesh feed sample should

produce a concentrate accounting for 1.41 weight percent of the feed. This concentrate will analyze 3.45 percent Ti, and contain 10.3 percent of the Ti values in the feed.

By changing the magnetic separator settings, a lower grade concentrate assaying 0.98 percent Ti can be projected. The Ti recovery will increase to 25.4 percent. The concentrate weight will be 11.7 weight percent of the feed.

Electrostatic, Magstream, and Magnetic Separator

Samples

The analyses of samples tested and their screen size distribution are summarized in Table 2.

Test Procedures, Equipment, and Results

Two samples of Millbillillie #173 meteoric eucritic material at -10 US mesh and -30 US mesh sizes were tested at the Carpco Laboratories in Florida, with the principal objective of beneficiating the ilmenite. Processes evaluated included the following:

- o Vertical Electrostatic (ES) free-fall separation
- o Electrostatic (ES) plate separation
- o Rare-earth drum magnet separation (KHD)
- o High-tension (HT) ES roll separation
- o High-intensity lift roll magnet separation
- o Magstream gravity concentration

The equipment, flowsheet, and procedures used in the test program is contained in Carpo's report, and will not be discussed here. The Carpco report is attached as Appendix 1. All products were returned to the University of Arizona for detailed analyses by the principal investigator.

A total of twelve tests were completed. The flowsheet details of each of the tests can be found in Carpco's report in Appendix 1. All of the analytical data is compiled in Appendix 3. A summary of the test results is presented in Table 4.

Discussion of Results

Based on visual and binocular optical microscopic examinations during the course of the test program and the test results given in Table 4, the following conclusions are possible:

- o The -30 mesh sample responded poorly to ES free-fall, ES plate, and rare-earth drum magnet separation processes. This was due to the lack of liberation within the sample, and

TABLE 3

Summary of Frantz Magnetic Separation Data on Millbillillie Sample

Test #	Vibrator Setting	Forward Slope (o)	Side Slope (o)	Particle Mesh Size	Final Product* Wt% of Feed	Final Product* Wt% of Size	Final Product* Analyses (%) Tot. Fe	Fe^{+2}	Ti	Final Product* Distribution (%) Tot. Fe	Fe^{+2}	Ti	Calcd Head (%) Tot. Fe	Fe^{+2}	Ti
T-2-1	6-4 AMP	15°	20°	-30 + 100	7.40	21.52	24.4	19.4	0.25	32.8	30.3	16.4	16.0	13.8	0.33
T-2-1-2	10	30°	20°	-30 + 100	7.73	22.46	16.3	13.6	0.37	25.4	24.4	22.0	14.4	12.5	0.38
T-1-1	6	15°	20°	-30 + 100	0.87	2.54	26.1	18.2	0.24	3.2	2.9	2.1	21.1	15.9	0.28
					33.71	97.98**	21.4	16.2	0.30	99.6	99.8	99.3	21.3	15.9	0.28
T-2-2	10	30°	20°	-100 + 200	6.97	34.52	24.2	19.9	0.36	50.8	49.3	38.4	16.4	13.9	0.34
T-1-2	6	15°	20°	-100 + 200	0.65	3.23	26.8	16.2	1.21	4.0	3.2	11.5	21.9	16.5	0.34
					18.89	93.49**	23.3	17.5	0.36	99.5	100.0	99.1	21.9	16.5	0.34
T-2-3	10	30°	20°	-200 + 400	4.05	22.64	25.8	20.2	0.98	36.4	31.7	52.4	16.0	14.4	0.42
T-1-3	6	15°	20°	-200 + 400	0.49	2.72	27.8	21.8	3.45	5.1	5.0	21.2	15.0	11.9	0.44
					13.44	75.07**	18.5	15.1	0.63	92.4	95.8	93.1	15.0	11.9	0.44
From PR Report 1		25°	15°	-30 + 400	44.59	61.50	17.7	15.4	0.36	66.4	68.1	62.9	16.3	13.9	0.35
Head Assay				-30 + 100		34.4	16.2	13.8	0.34	38.7	37.8	30.8			
Head Assay				-100 + 200		20.2	16.6	14.6	0.35	23.3	23.4	17.9			
Head Assay				-200 + 400		17.9	14.4	12.4	0.43	17.9	17.6	20.5			
Head Assay				-400		11.4	10.5	9.7	0.44	20.1	21.2	30.8			

*0.35 AMP Product

**0.35 x 1.25 AMP Product

TABLE 4

Summary of Electrostatic and Magnetic Separation Data on Millbillillie Sample

			<----------Final Product---------->								
			Analyses (%)			Distribution (%)			Calcd Head (%)		
Test #	Process	Particle Mesh Size	Wt%	Tot. Fe	Fe^{+2}	Ti	Tot. Fe	Fe^{+2}	Ti	Tot. Fe	Fe^{+2}	Ti
1	Single Pass, High TensionES	-30 mesh (100% of feed)	1.05	16.90	10.00	0.65	1.3	0.8	1.9	13.5	13.0	0.36
2	ES free fall separation	-30 mesh (100 % of feed)	<----------products not assayed---------->									
3	Screen sizing and distribution	only -400 mesh analyzed										
3A	Screen size distribution after grinding	-200 mesh product								15.1	13.1	0.36
4	Single pass ES	-30 mesh -- Products to Test 5										
5	ES -2 pass conductor retreat	-200 + 400 mesh (12% of -30 mesh sample)	5.56	19.05	15.80	2.73	7.6	7.1	39.1	14.0	12.3	0.39
6	Screen sizing and grinding											
7	ES-2 pass non-conductor retreat + Lift Magnetic Separator	-200 + 400 mesh (33.2% of -200 mesh sample)	5.44	20.10	18.70	0.74	7.1	7.0	10.5	15.5	14.6	0.39
8	ES-2 Pass non-conductor retreat	-200 + 400 mesh (51.9% of -200 mesh sample)	30.29	18.20	16.60	0.60	32.1	32.3	52.1	17.2	15.6	0.35
9	Screen for Magstream tests	-30 mesh	----------							13.7	12.8	0.39
10	Magstream spn. --S. G. 4.25	-200 + 400 mesh (12.0% of -30 mesh sample)	14.77	21.5	20.30	0.94	23.5	24.2	33.2	13.3	12.4	0.42
11	Magstream spn. -- S. G. 4.25	-30 + 200 mesh (72.7% of -30 mesh sample)	4.35	21.25	18.40	0.34	6.1	6.2	4.1	15.2	13.0	0.36
12	ES + Magstream sepn. of conductors from Test 8 at S. G. 4.25	-200 + 400 mesh (conductors were 15.7% of -200 mesh sample)	46.11	22.60	20.20	0.73	57.6	63.9	60.5	18.1	14.6	0.56

the presence of fines (Tests 1,2,and 3). The free-fall ES separator appeared to be effective in separating the fines from the coarse sizes. It could be useful in dedusting lunar samples.

o A single pass ES of the -200 + 400 fraction of the -30 mesh sample produced a conductor with 4 weight percent of the feed to the test in Test 4. After visual examination of the products under the microscope, the products were recombined for the two-pass ES test described below.

o The most significant physical upgrading was achieved in a Two-pass High Tension Electrostatic Roll Separator. The feed to the test consisted of the 200 x 400 mesh fraction of the -30 mesh sample. The 200 x 400 mesh fraction constituted 12 percent of the sample assayed 0.42 percent Ti, and contained 13.3 percent of the Ti values as shown in Table 2. The conductor material from the first pass was retreated in a second stage to produce a concentrate analyzing 2.73 percent Ti, with 39 percent Ti recovery. The two-pass conductor retreat product accounted for 5.44 weight of feed to the test, or 0.67 percent of the original -30 mesh sample, as shown in Test 5 results in Table 4. It is most probable that a single pass ES separation was just as effective as the two-pass ES test, since the conductor weight percentages were comparable on both tests.

o By projecting the above results to a -200 mesh grind, a two-pass ES conductor retreat product accounting for 2.88 weight percent of the total feed analyzing 0.39 percent Ti should be possible. This product is expected to analyze 2.73 percent Ti, with 18.9 percent recovery of the Ti values in the total feed sample.

o Attempts to improve ilmenite recovery by scavenging ilmenite values by using a ES two-pass non-conductor retreat method were not effective, as can be seen from the results of Test 8. The increased Ti recovery of 52 percent was gained at the expense of a lower grade of 0.74 percent Ti. Magstream separation of the conductors from Test 8 at an apparent specific gravity (S. G.) of 4.25, upgraded the composite conductor only slightly from 0.60 percent Ti to 0.73 percent in the heavies.

o Magstream separation did not improve the grade of the ES conductor product in Test 7.

o Magnetic separation of the ES two-pass conductor retreat product upgraded the Ti only marginally from 0.60 percent Ti to 0.74 percent Ti, with significant Ti recovery losses.

o Magstream gravimetric separation of -30 + 200 screen fraction at an apparent S. G. of 4.25 was not effective in Test 11 in upgrading the heavy fraction, due to the presence of unliberated ilmenite grains at this particle size. By contrast, the heavy fraction from the -200 + 400 screen fraction (with a significant proportion of liberated ilmenite grains) was upgraded from 0.42 percent Ti to 0.94 percent Ti with 33.2 percent Ti recovery in a concentrate representing 14.8 weight

percent of the feed to the test, contained 19.3 percent of the Ti values, with an assay of 1.37 percent Ti. The combined product would be 20.7 weight percent, with an analysis of 1.06 percent Ti, and 52.6 percent Ti recovery. The metallurgical efficiencies and recoveries of the combined products would be comparable to the previous Magstream test data reported earlier in Reference 1.

o The above results can be projected to a -200 mesh grind sample, containing 51.9 percent as -200 + 400 mesh, and 48 percent of the Ti values. Magstream separation would thus produce a heavy concentrate with 10.74 weight percent, and 27.3 percent recovery at a grade of 1.06 percent Ti. It would be interesting to determine if ES roll separation of this heavy fraction would improve grade with minimum recovery losses.

Conclusions

o Dry magnetic separation in a Frantz Isodynamic Separator was effective at 200 mesh and finer sizes. It should be possible to produce a concentrate analyzing between 1 percent Ti and 3.5 percent Ti, corresponding to Ti recoveries between 25.4 percent and 10.3 percent. The concentrate weight will be between 11.7 percent and 1.4 weight percent of the feed. The feed to the test was 0.39 percent Ti.

o A two-pass dry Electrostatic Roll Separator produced a concentrate analyzing 2.73 percent Ti, with 18.9 percent Ti recovery. The concentrate weight percent was 2.9 percent of the feed.

o Wet gravimetric-magnetic separation in the Magstream apparatus was successful in producing a concentrate analyzing 1.06 percent Ti with a concentrate weight of 14.8 percent of feed and Ti recovery of 52.6 percent.

o The vertical V-STAT Electrostatic free-fall separator appeared to hold promise for dedusting samples.

Recommendations

Future work along the following lines is recommended on the Eucrite Sample:

o Wet Magstream, followed by dry ES roll separation of the heavy fraction.
o Dry Magnetic separation, followed by dry ES roll separation.
o Test V-Stat separator for efficiency of dedusting.
o Complete SEM and EDS scans of concentrates produced from tests with the ES two-pass separator, and the Frantz Magnetic Separator.

The additional data to be generated on the eucrite will determine the scope of process engineering, and scientific studies on the very limited quantities of lunar material (5 to 10 grams) to be made available in 1993.

References

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