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Mineralogical Characterization of an Ilmenite Ore Sample and the Concentration of Titanium

THESIS – MSE499Y

by
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Abstract

This thesis investigated the mineralogy of an ilmenite ore sample and used gravity separation and magnetic separation to make a titanium concentrate using different grind sizes. The objective was to develop the best titanium concentrate grade possible. The concentration techniques chosen were gravity separation and magnetic separation based on the test work done by the Quebec Mining and Titanium Corporation on a similar ore type.

The sample was analyzed by XRF, ICP, XRD and optical microscopy to determine the chemical composition, mineral composition and mineral associations.

The sample was crushed and divided into size fractions that were used in the separation tests to determine the best particle size for the concentration techniques. The size fractions were examined for liberation by optical microscopy.. Composition of the fractions was found by XRF. The gravity separation was done by using methylene iodide as the medium in heavy liquids separation. Magnetic separation was done by a rare-earth magnetic separator.

The mineralogical analysis found that there was hematite present throughout the ilmenite grains and that it would not be possible to separate the two by physical means. The sample was homogeneous and composed of approximately 20% titanium dioxide. Gravity separation by heavy liquids removed the silicates prior to magnetic separation. Magnetic separation concentrated the titanium in the magnetics portion. The best grade achieved was 33.4% titania with particles sizes of less than 300μm.

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Chapter 1 INTRODUCTION

Titanium is a highly desirable metal due to its many useful physical properties. It is hard, has a high melting temperature, is lightweight and is very corrosion resistant. It is as strong as steel, but is 45% lighter, and twice as strong as aluminum. These physical properties make titanium and titanium alloys very useful in the aerospace industry where materials need to withstand extreme temperatures and have a high strength to mass ratio. The aerospace industry accounts for 60% of the metallic titanium market. Examples of aerospace applications are: engines; framework for spacecrafts; and construction materials for space stations [1].

Titanium dioxide has many applications in variety of areas because of its unique qualities. Accounting for 94% of the titanium market, the most popular use for titanium is pigment grade titania. Titanium dioxide is second to diamond for its refractive index and is used for whitening paints, plastics, paper, and rubber. Titania is also used as an opacifier in glass and porcelain enamels, cosmetics, and sunscreen. Another use for titania is as a photosensitiser for photovoltaic cells, and when used as an electrode coating in photoelectrolysis cells can enhance the efficiency of electrolytic splitting of water into hydrogen and oxygen. The photocatalytic activity of titania results in thin coatings of the material exhibiting self cleaning and disinfecting properties under exposure to UV radiation. These properties make the material a candidate for application in medical devices, food preparation surfaces, and air conditioning filters [2].

The two most important titanium bearing minerals are ilmenite ($FeTiO_3$) and rutile (TiO_2). Ilmenite provides 90% of the titanium used every year. It is estimated that world resources of ilmenite contain 1 billion tons of titanium dioxide. The estimations for the titanium dioxide content in rutile is 230 million tons [2].

The objective of this thesis is to make a titanium concentrate of the highest grade possible through physical means from an ilmenite ore sample.

Chapter 2 BACKGROUND

Canada has several large deposits of ilmenite in Quebec. The Quebec Iron and Titanium Corporation (QIT) has continuously been mining one of these deposits for ilemnite since the 1950's. The sample being investigated originated from a site next to the QIT operation, the Allard Lake titanium deposit. The QIT deposit is thought to have similar ore characteristics as the sample being investigated.

3.1 Anticipated Ore Characteristics and Composition

According to Elliot [3] the anticipated ore is ilmenite-hematite, where the ilmenite is closely associated with the iron oxide hematite. The ore has exsolved hematite lamellae and in the host material, ilmenite. The hematite lameallae in many cases traverses the whole grain of ilmenite and may range in width from 100 microns down to small needles, barely visible under the high-power objective. This intimate relationship between the minerals make it appear as though it would be impossible to separate the two by any mechanical means. Gangue mineral associated with this ore are mainly plagioclase feldspars and in smaller proportions apatite, hypersothene (a magnesium iron silicate), and mica [3].

3.2 Concentration Methods Investigated and Practiced by QIT

The Lac Tio ore mined by the QIT Corporation is very coarse grained and the liberation of ilmenite-hematite from gangue is almost complete at 635 microns. The methods investigated for concentration were: flotation; gravity separation; electrostatic separation; and high-intensity magnetic separation. The flotation results by either cationic or anionic flotation on minus 300 microns material yielded concentrates ranging from 70% for the high-grade feed to 50% for the lower-grade feed. Gravity concentration by tabling gave concentration grades of approximately 90% combined ilmenite-hematite, with recovery of about 90%, this operation was determined to be too costly to be practical. Electrostatic separation was found to be successful for up-grading the fines only when the feed to the separator was similar in size, this operation was also considered to be too costly to be practical. High-intensity magnetic separation gave the best metallurgical results of any method tested [3].

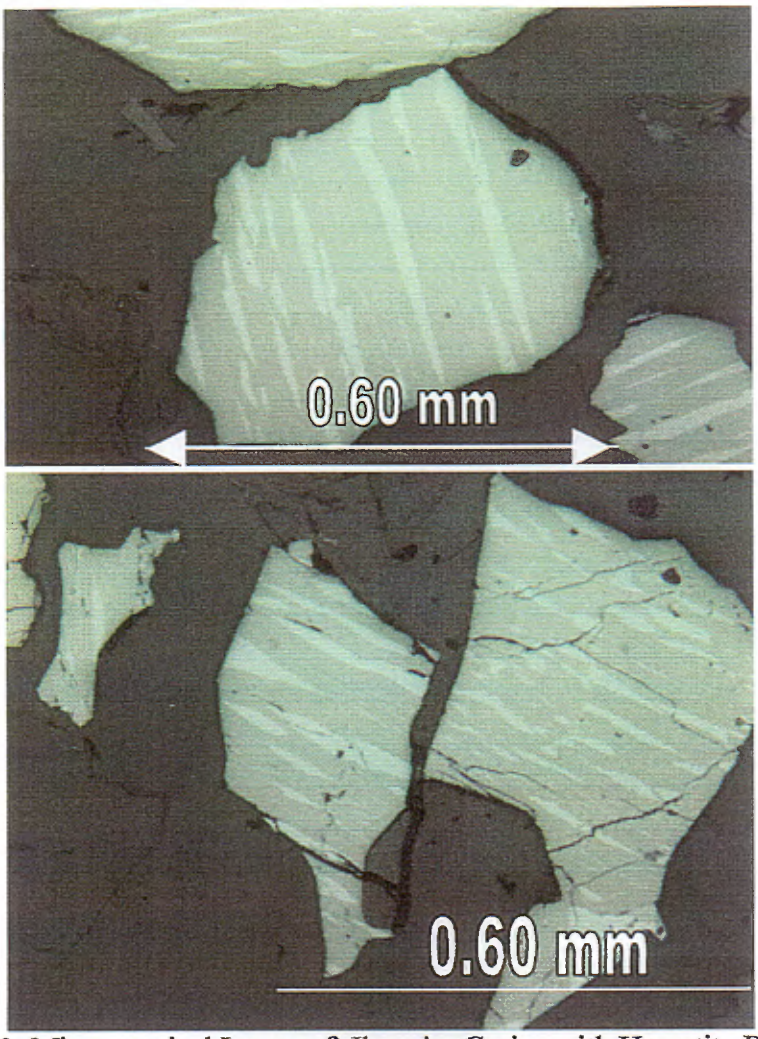


Figure 2. Microscopical Image of Ilmenite Grains with Hematite Blades

Table 3. Screen analysis of bulk sample.

Sample	Mesh	Microns	Weight %	Cumulative	Weight %
Size 1	12	1700	0.5	100.0	20.1
	16	1180	19.6	99.5	
Size 2	20	850	17.9	79.9	17.8
Size 3	30	600	17.2	62.1	17.2
Size 4	40	425	13.1	44.9	22.6
	50	300	9.5	31.8	
Size 5	70	212	7.9	22.3	22.3
	100	150	4.7	14.3	
	150	106	3.7	9.6	
	200	75	2.2	5.9	
	270	53	1.4	3.8	
	400	38	0.9	2.4	
	pan	-38	1.5	1.5	

It was found that the best separation methods were high-intensity magnetic separation and gravity separation. Electrostatic separation and flotation were rejected as concentration methods for this ore [3].

CHAPTER 3 EXPERIMENTAL

The experiments and analyses were done at Lakefield Research except for a few reflected light microscopy pictures, which were taken at the University of Toronto.

3.1 MATERIAL

A 4.8 kg sample of ilmenite ore was obtained from SGS Lakefield Research Ltd.. It was composed of several heavily oxidized pieces ranging in weight from 300g to 1100g (Figure 3-1).

Figure 3-1: Sample of ilmenite ore as received from SGS Lakefield Research Ltd.

3.2 EQUIPMENT AND INSTRUMENTS

Equipment used for sample preparation was: a jaw crusher; a Ro-tap with mesh sizes 12, 16, 20, 30, 40, 50, 70, 100, 150, 200, 270, 400; a pulverizer, and rifflers. Three sizes of rifflers were used to accommodate different amounts of material. They were, listed from largest to smallest: a rotating riffler; a slot riffler; and a rotating micro riffler.

Two instruments were used for whole rock analysis. The first was a Bruker 3400 XRF spectrometer, the samples were prepared by fusion in a Claiss Fluxy. The second was a Radial Varian Vista ICP spectrophotometer. Instruments used for ore characterization were an optical microscope with an integrated digital camera; and a Siemens D5000 diffractometer for X-Ray Diffraction (XRD). Methylene iodide with a density of 3.1g/cm^3 was used as the heavy medium for gravity separation by heavy liquids. The equipment set up for gravity separation is shown in section 3.3.3.A (Figure 3-8). A rare-earth magnetic separator (section 3.3.B Figures 3-9 and 3-10) was used for magnetic separation. See Appendix A for pictures of equipment and instruments.

3.3 EXPERIMENTAL PROCEDURE

The experimental procedure was divided into three main sections:3.3.1 Bulk Sample Preparation and Compositional Analysis; 3.3.2 Particle Size Classification and Compositional Analysis; and 3.3.3 Concentration of Titanium. The sub sections detail the sample preparations, tests performed, and any analyses that were performed. See Figure 3.2 for an overview of the experiments performed.

Experimental Procedure

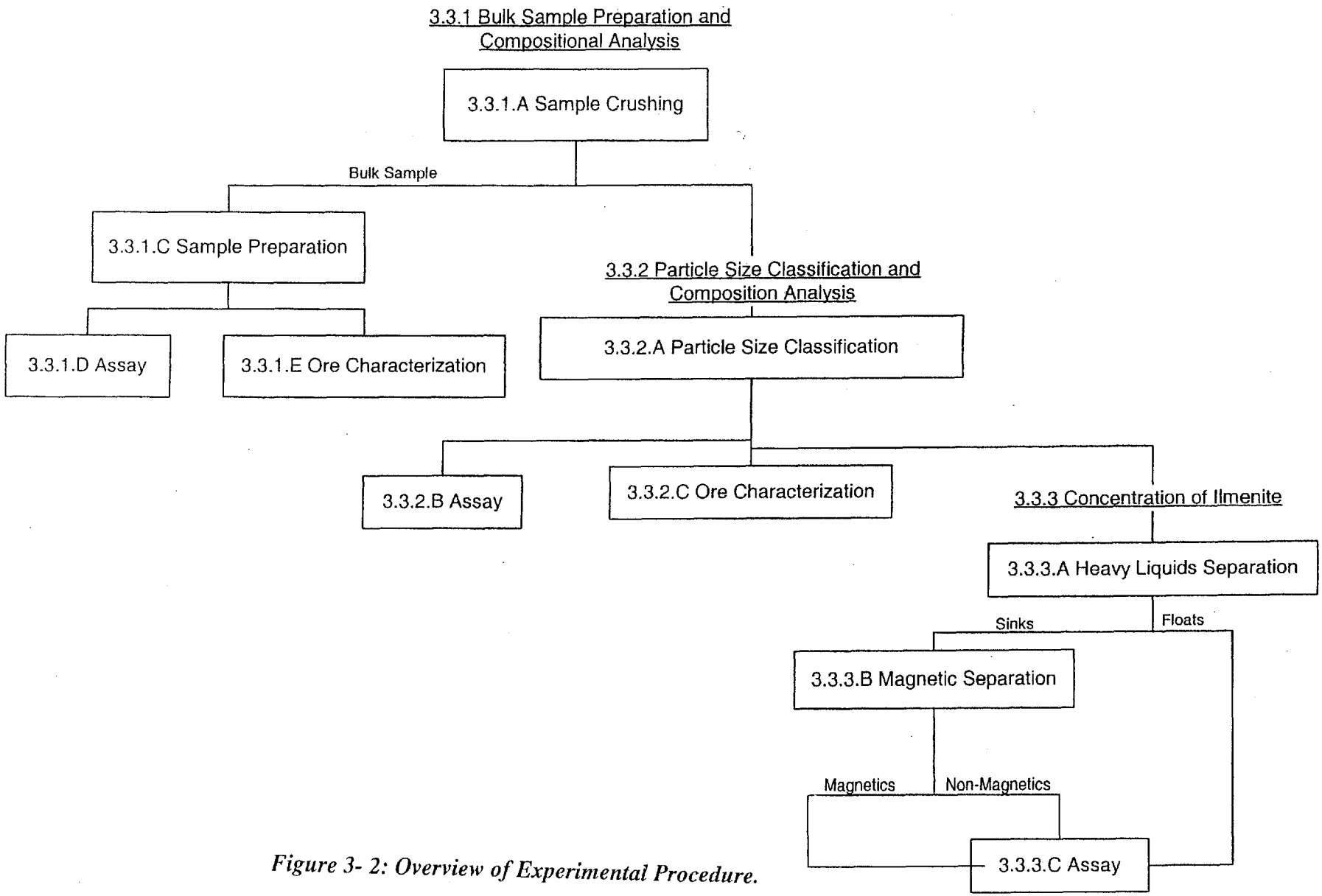


Figure 3- 2: Overview of Experimental Procedure.

3.3.1 Bulk Sample Preparation and Compositional Analysis

This section established the nature of the bulk sample by compositional, elemental and mineralogical analyses.

3.3.1.A Sample Crushing

A jaw crusher was used to reduce the 4.8kg of material to a particle size of less than 1.7mm. Figure

3-3 shows how the sample was then divided into 12 representative portions by a rotating riffler. The portions were then combined in varying proportions to make three groups:

-A (1/12) was for bulk sample compositional analysis

-B (8/12) for particles size classification

-C (3/12) was retained as a spare for further analyses.

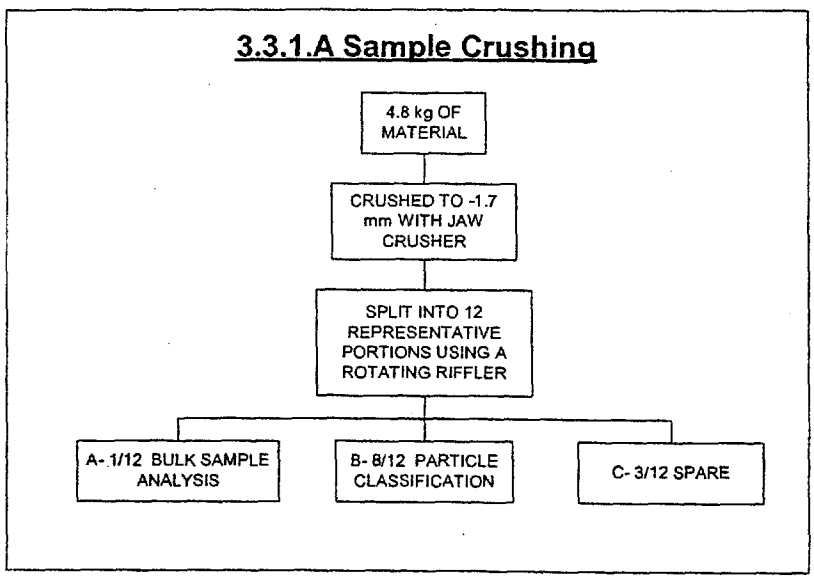


Figure 3-3: Process flow diagram for sample crushing

3.3.1.B Sample Preparation

Group A was divided into 3 representative portions:

-A.1 (¼) for XRD, XRF, ICP (Figure 3-4)

-A.2 (¼) for optical microscopy

-A.3 (½) was retained as a spare for further analyses.

The analysis methods for group A.1 (XRD, XRF, and ICP) required the sample to be homogenous and have a fine particles size between 5 and 10μm. The necessary particle size was achieved by grinding the sample in a pulverizer for around ~40 sec.

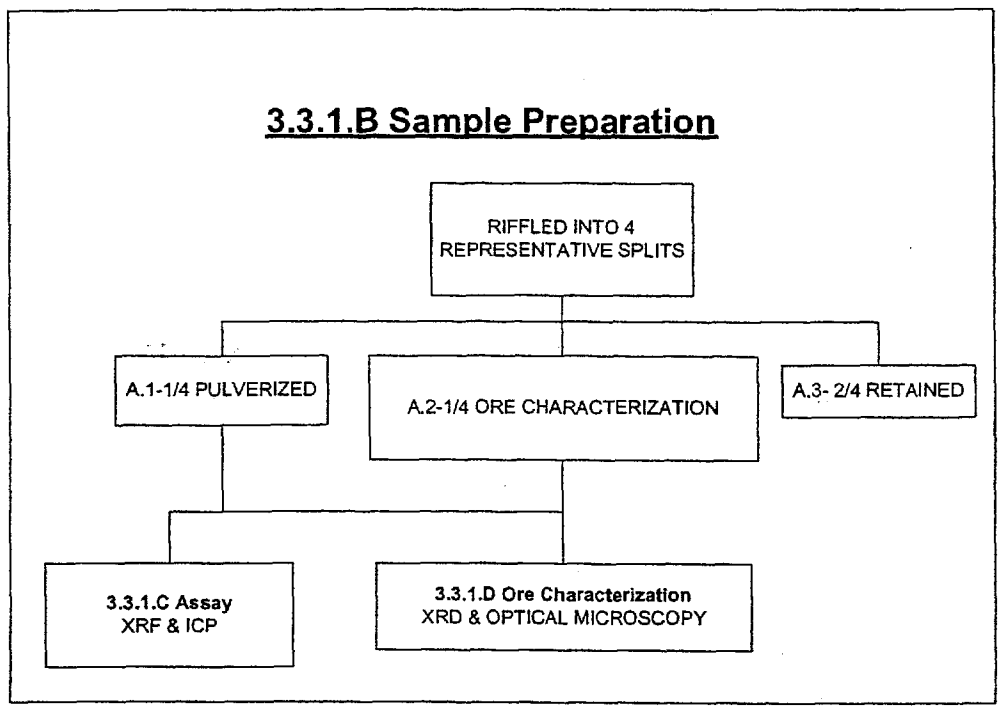


Figure 3-4: Flow diagram for compostional analysis sample preparation.

3.3.1.C Assay

Whole rock analysis by XRF required mixing 0.5g of the powered sample (A.1) with 5.0g of lithium metaborate. This mixture was dumped into a platinum crucible and 1ml of ammonium nitrate was added. The lithium metaborate was the matrix for the sample. Lithium metaborate was used as the matrix because it was used in the detection of x-rays and would not interfere with the detection of the other compounds. The

ammonium nitrate was used as an oxidant to ensure the complete oxidation of the elements. The crucible was mounted in the automatic fuser, fused for 2 minutes, poured into a disk and cooled quickly. This made a solid homogeneous glass disk approximately 3cm in diameter, that was placed in the XRF spectrometer. Lost on ignition (LOI) was found by placing one gram of each powdered sample in a furnace at a temperature of 1010°C for 1 hour. The sample was then weighed to calculate the amount of material lost on ignition. The LOI number was used to give a general indication of the "volatile" species in the sample so that the results given by the spectrometer could be quantified accurately [5].

Whole rock analysis by ICP requires approximately 1g of the powdered sample. The sample underwent a 4 acid digestion with hydrochloric, nitric, hydrofluoric and perchloric acids, and brought up in a final matrix of 20% hydrochloric acid. The sample solution was then placed into the core of an inductively-coupled plasma at a temperature of approximately 8000°C. The spectrometer recorded the emitted light and converted the information to elemental concentration [4].

3.3.1.D Ore Characterization

The major minerals present were determined by XRD. Approximately 2g of the powdered sample (A.1) was placed in the diffractometer to be analyzed. The scan conditions were Co Radiation, graphite monochromator, 40Kv, 30mA, steps of 0.02° with a step time of 1 second. The detection limit was 0.5-2% and was strongly dependent on crystallinity.

Group A.2 was riffled into 3 portions by a micro rotating riffler with 8 compartments (Figure 3-5):

-1/8 was made into a polished thin section

-1/8 made into a polished section

-6/8 were retained for as a spare for further analyses.

Optical microscopy was performed to identify minerals below the detection limit of the diffractometer and to determine the associations between the minerals. The thin polished section was examined by transmitted light and the polished section was examined with reflected light.

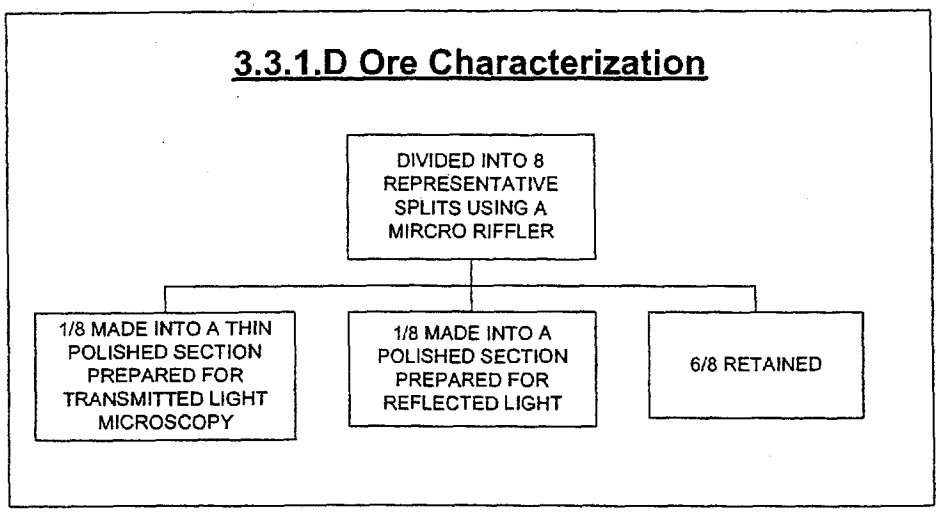


Figure 3-5: Process flow for dividing sample for ore characterization.

3.3.2 Particle Size Classification and Compositional Analysis

In this section different particle sizes were examined by chemical and microscopic means. This was done to note any differences in mineral liberation and chemical composition at the different particle sizes.

3.3.2.A Screen Analysis

Particle size classification was performed on group B of the original bulk sample by a Ro-tap. The Ro-tap had 12 screens of different mesh sizes and was set to vibrate for 15 minutes. It divided the sample into 13 size fractions: +1700, +1180, +850, +600, +425, +300, +212, +150, +106, +75, +53, +38, -38μm. Each fraction was weighed to find the particle size distribution. The 13 fractions were then combined to make 5 classes of particle sizes of approximately the same weight. The samples were labeled composite 2 through 6 where composite 2 (comp 2) was composed of the largest particle sizes and comp 6 was the smallest (see Table 4-5 in Results section 4.4.2). The different groups of particle sizes were used in the subsequent tests to determine the optimum particle size for the concentration of titanium (see Figure 3-6).

The composite samples were divided for analysis. They were riffled into representative splits and combined to make groups:

-D (2/10) for assay

-E (1/10) for ore characterization

-F (1/10) for concentration of ilmenite

-G (6/10) retained for spare samples and further analysis.

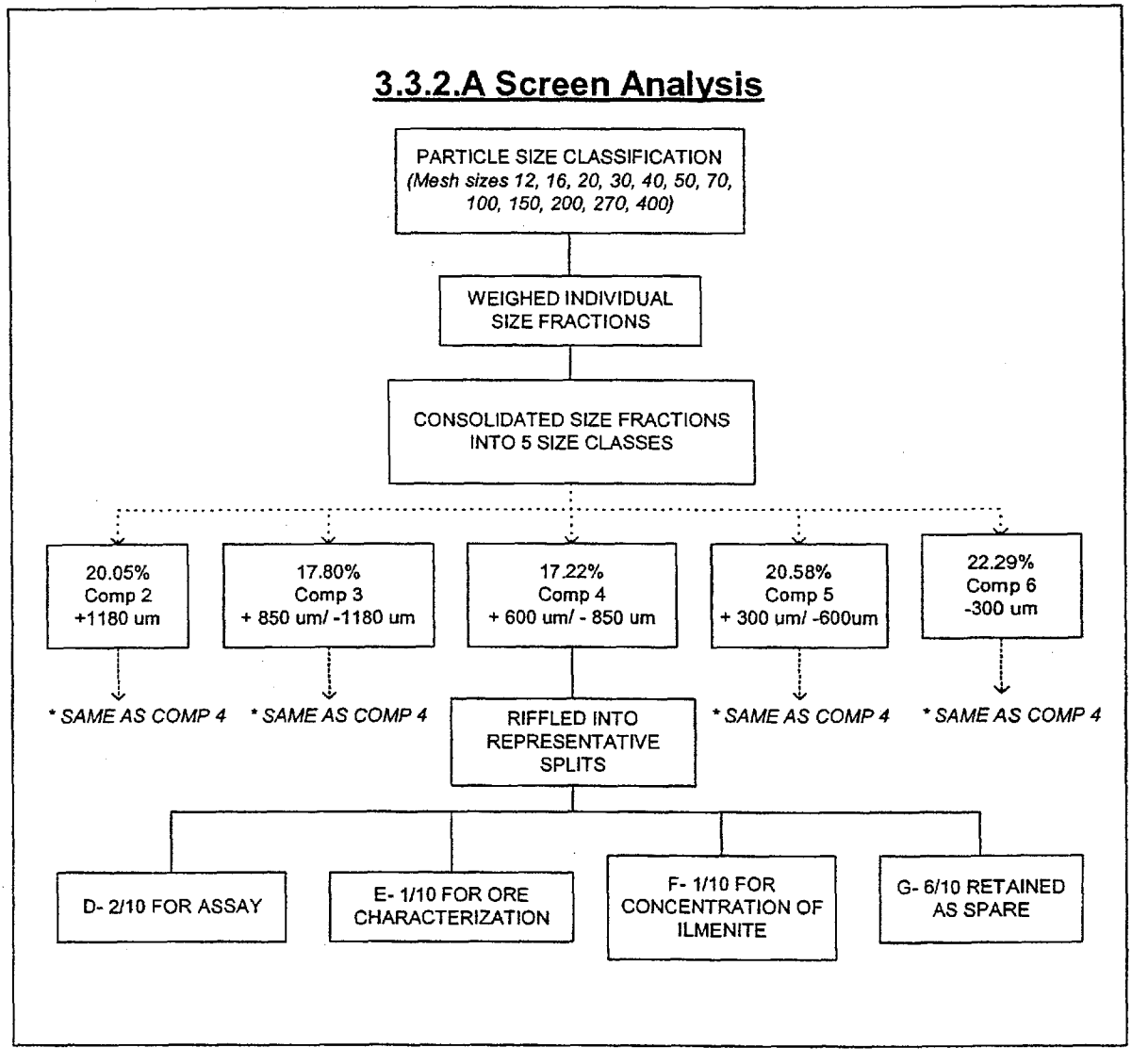


Figure 3-6: Flow diagram showing screen analysis, consolidation of size factions and division of samples for analysis.

3.3.2.B Assay

Group D of each composite sample was then pulverized for whole rock analysis by XRF according to the method described in section 3.3.1.C.

3.3.2.C Ore Characterization

Group E of each composite was riffled into 3 representative portions (Figure 3-7):

-E.1 for loose particle characterization by optical microscopy

-E.2 for polished section preparation and

-E.3 retained for future work if necessary.

The sample E.1 of each composite 2-6 was placed in a petri dish and examined for mineral associations with light microscopy. Samples E.2 were made into thin polished sections using a 2-part cold setting epoxy resin. The polished sections were then examined by reflected light microscopy. Digital pictures were acquired of the samples.

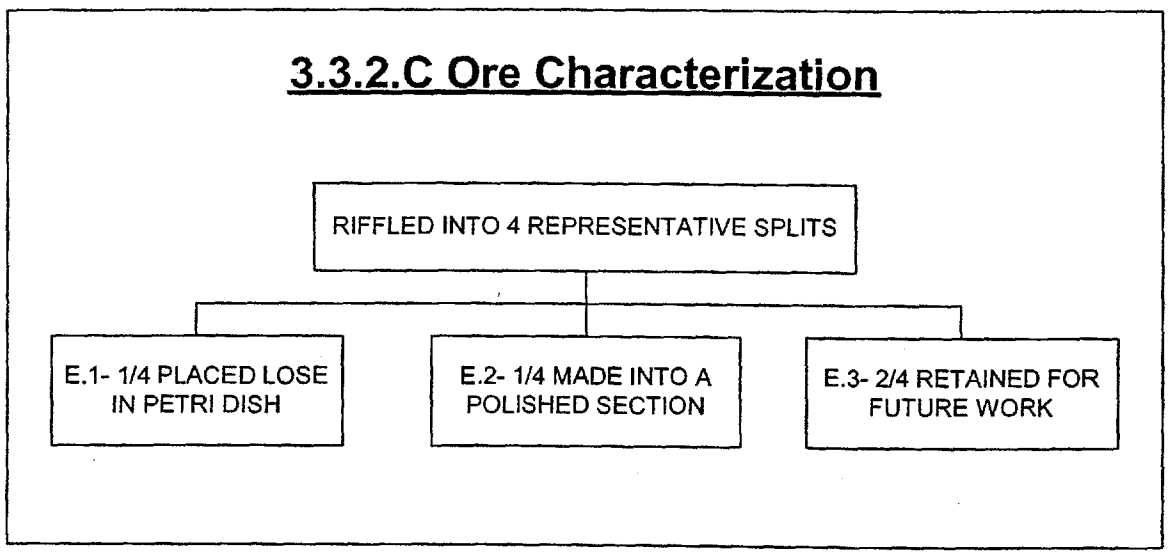


Figure 3-7: Flow diagram showing division of sample for ore characterization.

3.3.3 Concentration of Titanium

3.3.3.A Heavy Liquids Separation

Heavy liquids separation was performed on Group F of each composite sample, experimental set up can be seen in Figure3-8. The medium used in the heavy liquids separation was methylene iodide, it had a density of 3.1g/cm^3. The methylene iodide was used to separate the heavier minerals from the lighter silicates. The material was therefore divided into two portions; floats for light minerals and sinks for heavy minerals. The equipment set up can be seen in Figure 3-8. A retort stand supported a funnel that had a hose attached at the outlet, which was clamped closed. Methylene iodide was poured into the funnel and then the composite sample was added. The mixture was then stirred and allowed to settle then stirred again. Once the material showed no evidence of further settling the clamp was opened until the sinks and suspended particles had drained out and then the clamp was shut to retain the floats in the funnel. The sinks were collected in a beaker under the funnel. The material was therefore divided into two portions, sinks and floats. Two filter stations were set up, one for the methylene iodide and the other for the acetone that was used for a wash. The sinks were then poured into the filter, lined with filter paper, and the methylene was collected to be used again. The filter was then transferred to a second flask where the sinks were washed out of the beaker onto the filter paper and the particles were washed with acetone. The sinks were then dried in an oven. The same procedure was followed for the floats.

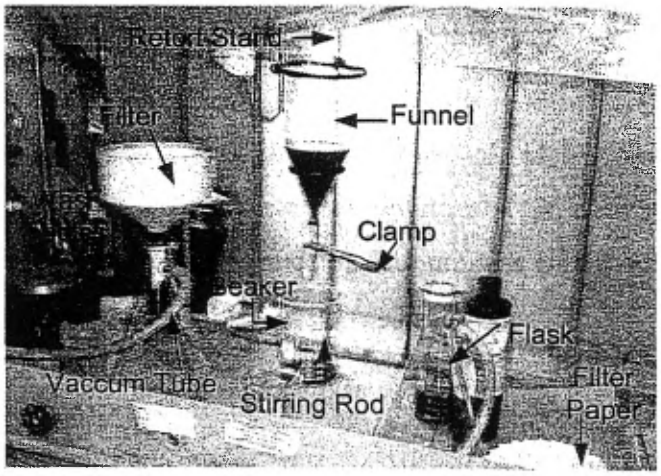


Figure 3-8: Experimental Set-up for Heavy Liquids Separation

3.3.3.B Magnetic Separation

The sinks from each composite sample were put through a magnetic separator of Gauss strength ~1200 (Figures 3-9 and 3-10). The material was divided into 3 bins; 2 were of the non-magnetic material and the other was for magnetics. The material from the two non-magnetic bins were combined and passed through the magnetic separator a second time. A hand held magnet of strength 500 Gauss was used to remove any magnetic material in the non-magnetic bins and placed in the magnetic bin. The hand held magnet was passed over the non-magnetic bin until no additional magnetics were being removed.

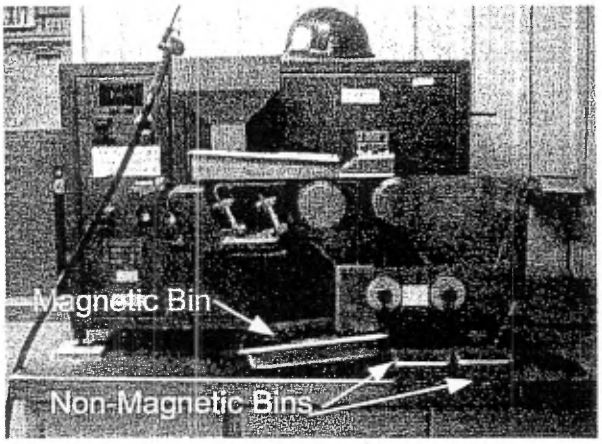


Figure 3-9: Experimental Set-up for Magnetic Separation

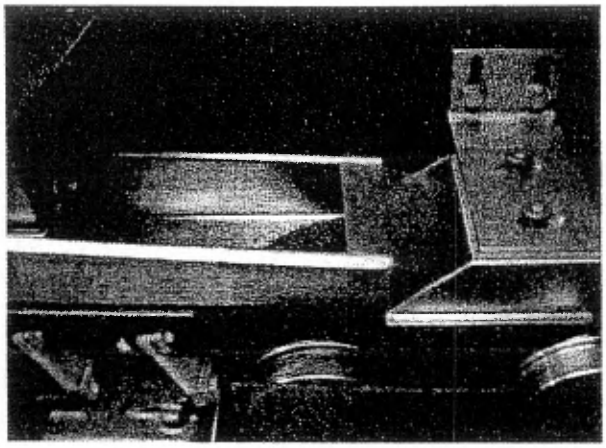


Figure 3-10: Top View of Magnetic Separation

3.3.3.C Assay

The floats, magnetics and non-magnetics of each composite sample were pulverized and analyzed by XRF as described previously. At each stage of separation the weights were recorded and a mass balance of titanium was calculated to obtain a recovery curve.

Chapter 4 RESULTS

4.1 BULK SAMPLE ANALYSIS

4.1.1 Elemental Composition Analysis

Assays were done by XRF and ICP as described in section 3.3.1.C. Assay by XRF (Table 4-1) found that the major oxides present were iron oxide, titania, and silica (Figure 4-1). Those oxides present in moderate amounts were alumina, magnesia, chromium oxide, and vanadium oxide. To determine minor constituents ICP was used. The results are given in Table 4-2 for the ICP analysis. The elements identified in bold are the ones not covered by the XRF analysis and are the values that should be observed for this test because ICP is unreliable for elements present in major amounts. The results for ICP (Table 4-2) are given in grams/ton therefore dividing by 10 000 gives the element composition in percent. The trace element present in the greatest amount was Co at 230g/t or 0.023%.

Table 4-1:Chemical composition of sample determined by XRF.

Sample ID	Fe2O3	TiO2	SiO2	Al2O3	MgO	CaO	NaO	P2O5	V2O5	k2O	MnO	Cr2O3	LOI	Sum
Comp 1	40.00	21.10	21.10	6.55	5.39	3.37	1.12	1.00	0.22	0.18	0.15	<0.01	-0.90	99.40

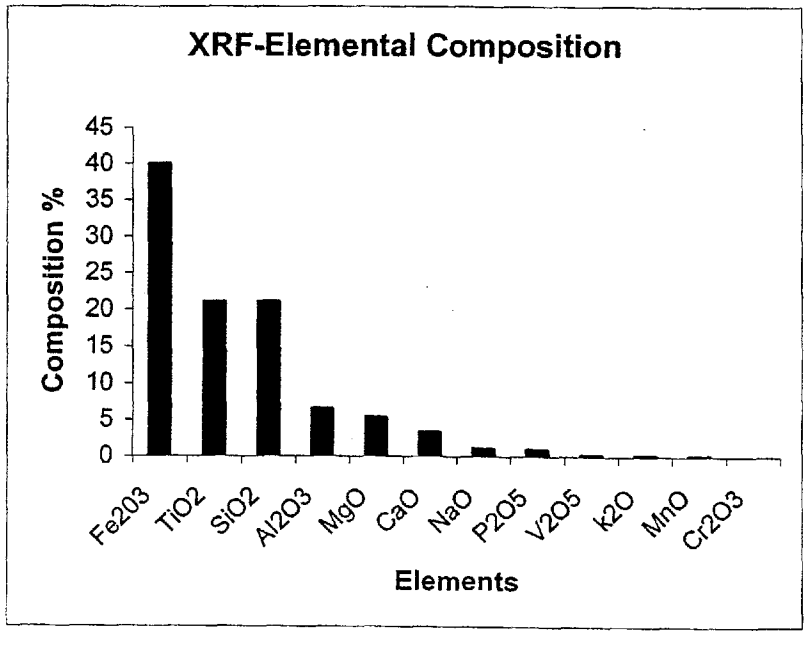


Figure 4-1: Graph showing chemical analysis results by XRF.

Table 4-2: Elemental composition analysis by ICP.

Sample ID	Ag	Al	As	Ba	Be	Bi	Ca	Cd	Co	Cr	Cu	Fe	K	Li	Mg	Mn
Comp 1 (g/t)	< 2	34000	< 50	78	< 1	< 30	25000	< 5	230	62	140	240000	1500	< 25	25000	730

Sample ID	Mo	Na	Ni	P	Pb	Sb	Se	Sn	Sr	Ti	Tl	U	V	Y	Zn
Comp 1 (g/t)	26	7000	88	3700	63	< 20	< 100	< 100	200	87000	< 100	< 75	940	4.3	64

4.1.2 Ore Characterization

Ore characterization was performed according to the method detailed in section 3.3.1.D. Analysis with XRD revealed that the major mineral present was ilmenite, moderate is plagioclase (feldspar), and minor minerals are hematite, magnetite, and pyroxene. The XRD pattern can be seen in Figure 4-2 and chemical composition for these minerals are in Table 4-4.

Table 4-3: Chemical composition of minerals identified by XRD.

Mineral	Composition
Hematite	Fe_2O_3
Ilmenite	$FeTiO_3$
Magnetite	Fe_3O_4
Plagioclase	$(NaSi,CaAl)AlSi_2O_8$
Pyroxene	$(Ca,Na)(Mg,Fe,Al,Ti)(Si,Al)_2O_6$

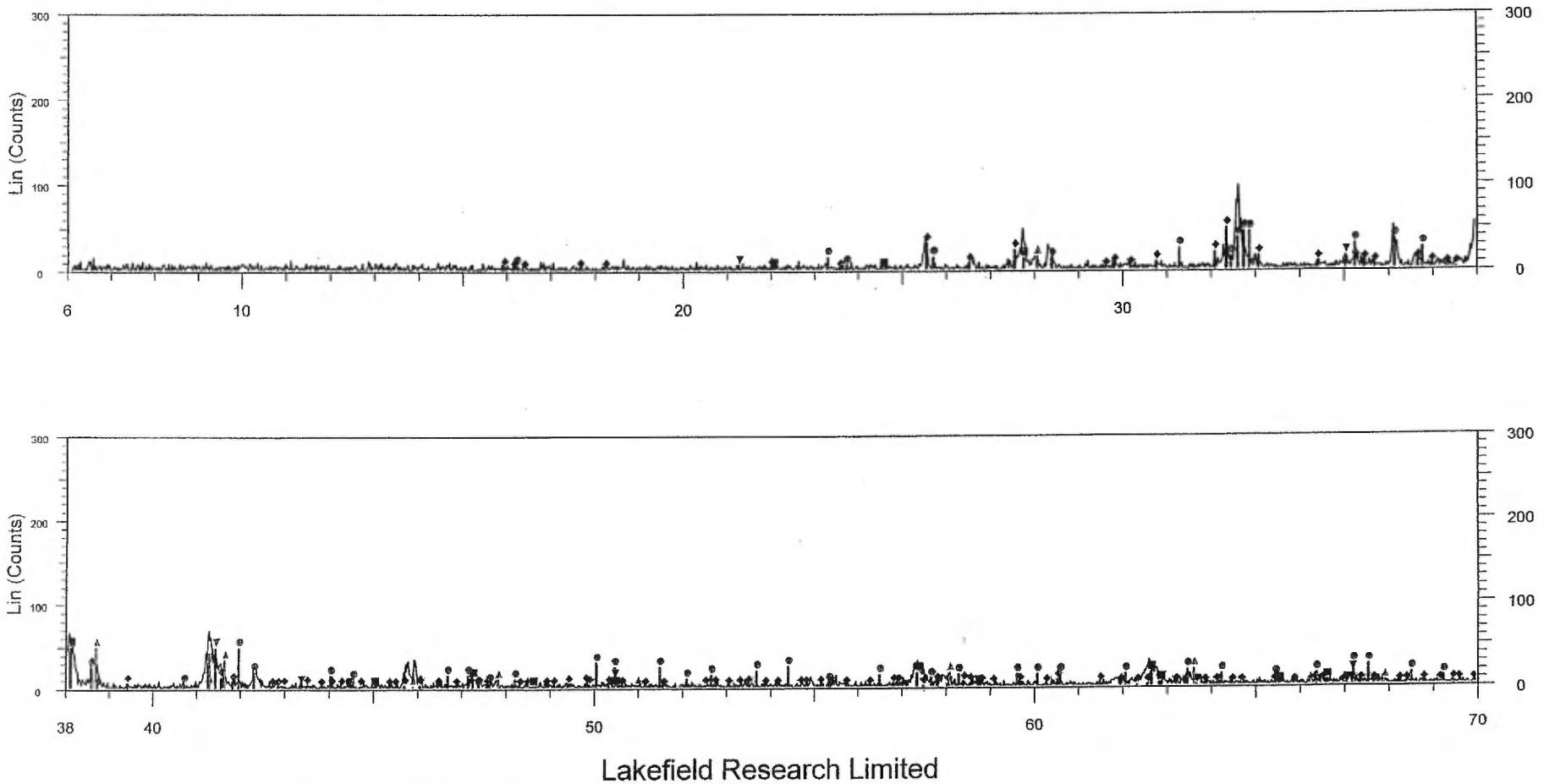


Figure 4-1: XRDPattern for Bulk Sample.

Optical microscopy confirmed the presence of the minerals identified by XRD. Several other minerals were also identified (Figures 4-3): pyrite (an iron sulfide); chalcopyrite (a copper sulfide); mica, muscovite, and quartz (silicates); goethite and rutile (oxides and hydroxides); and apatite (a phosphate). In most cases the hematite was present as a needle like structure distributed throughout the ilmenite grains. In a few cases the hematite had a speckled presence in the ilmenite grains.

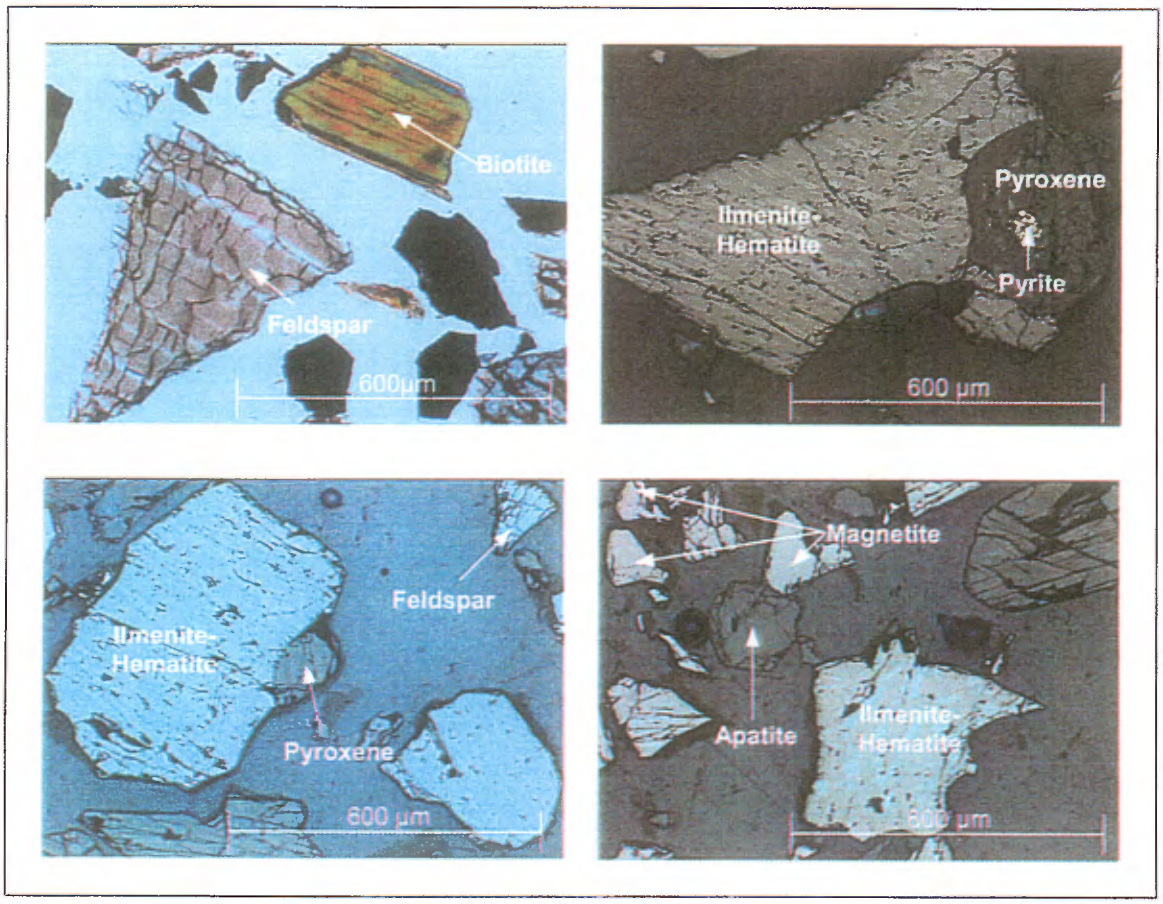


Figure 4-3: Minerals identified by optical microscopy. Top left is transmitted light and the other three are reflected light.

4.2 PARTICLE SIZE CLASSIFICATION AND COMPOSITION ANALYSIS

4.2.1 Particle Size Distribution

Particle size distribution was performed according to section 3.3.2.A. The cumulative passing weight percent can be seen in Figure 4-4. The size fractions were then consolidated into 5 composite samples of approximately the same weight (Table 4-5).

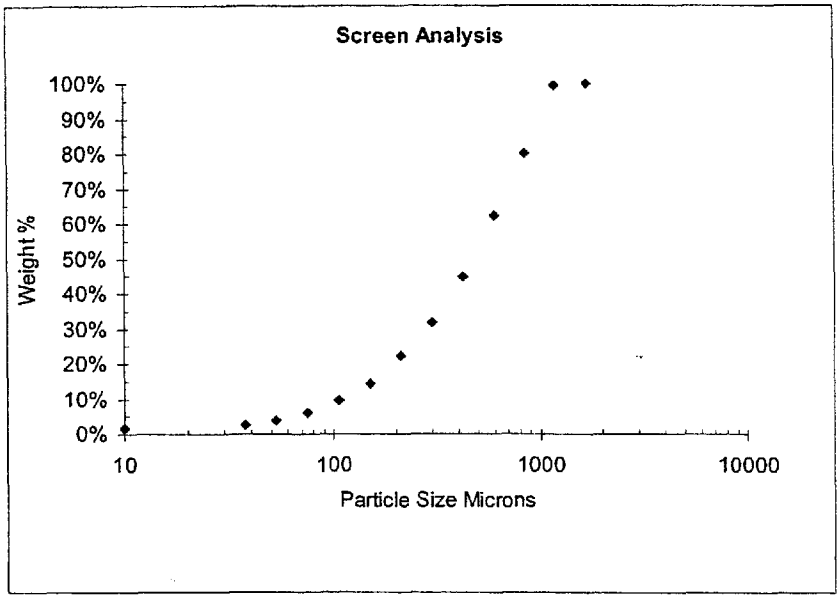


Figure 4-4: Particle size distribution showing cumulative passing of mesh size.

Table 4-4: Screen Analysis of Bulk Sample

Sample	Microns	Mesh	Weight (g)	Weight %	Cumulative %	Comp Cumulative %
Comp 2	1700	12	14.96	0.49%	100.00%	
	1180	16	594.32	19.56%	99.51%	20.05%
Comp 3	850	20	542.32	17.85%	79.95%	17.80%
Comp 4	600	30	523.44	17.23%	62.09%	17.22%
Comp 5	425	40	397.22	13.07%	44.87%	
	300	50	288.71	9.50%	31.79%	22.58%
Comp 6	212	70	241.37	7.94%	22.29%	
	150	100	142.91	4.70%	14.34%	
	106	150	112.47	3.70%	9.64%	
	75	200	65.71	2.16%	5.94%	
	53	270	41.84	1.38%	3.77%	
	38	400	28.32	0.93%	2.40%	
	-38	pan	44.5	1.46%	1.46%	22.29%
Total			3038.09			

4.2.2 Assay Comparison of Size Fractions

Chemical analysis was done by XRF. Table 4-6 shows that the elemental composition of the 5 composite samples was very similar.

Table 4-5: Composite Sample Analyses by XRF

Sample ID	Fe2O3	TiO2	SiO2	Al2O3	MgO	CaO	NaO	P2O5	V2O5	K2O	MnO	Cr2O3	LOI	Sum
Comp 2	39.20	20.80	23.00	7.02	5.67	3.05	1.23	0.60	0.22	0.22	0.15	0.01	-1.14	100.10
Comp 3	40.00	21.30	22.30	6.26	6.13	2.70	1.13	0.54	0.23	0.20	0.16	<0.01	-1.26	99.70
Comp 4	40.20	21.40	22.20	5.80	6.58	2.48	0.96	0.48	0.24	0.18	0.17	0.02	-1.23	99.40
Comp 5	40.40	21.60	21.70	6.49	5.77	2.73	1.10	0.51	0.24	0.19	0.16	0.02	-1.06	99.90
Comp 6	40.70	20.40	17.90	7.07	3.26	5.67	1.09	2.74	0.24	0.20	0.12	0.01	-0.12	99.20

4.2.3 Comparison of Liberation Achieved at Different Particle Sizes

Mineral liberation at each size classification was examined using optical microscopy of polished sections prepared from the composite samples (section 3.3.2.C). Pictures of the polished sections, and loose particles are compared in Tables 4-7 and 4-8.

Table 4-6: Mineralogical Comparison of Composite Samples 2 and 3.

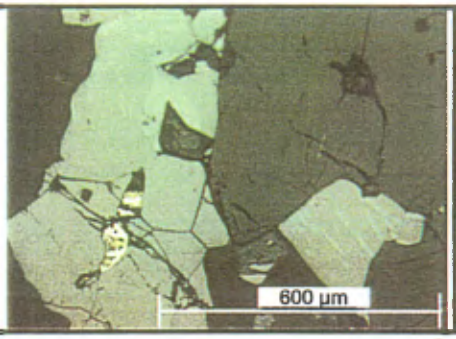
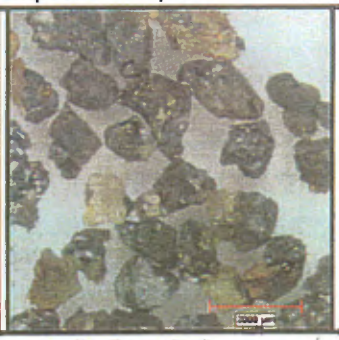
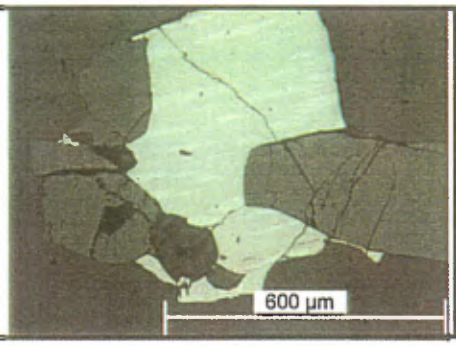
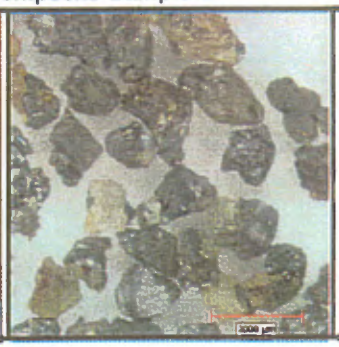
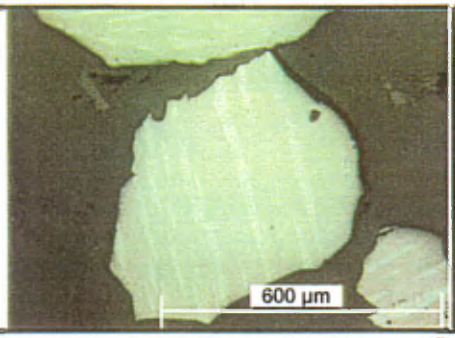
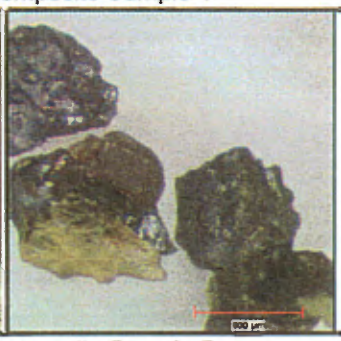
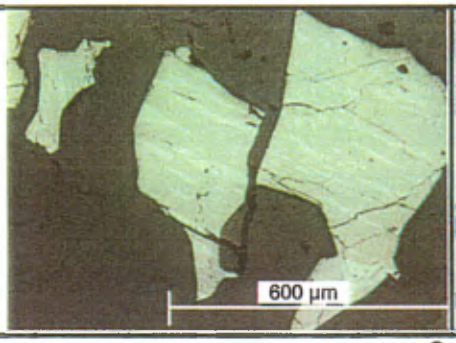
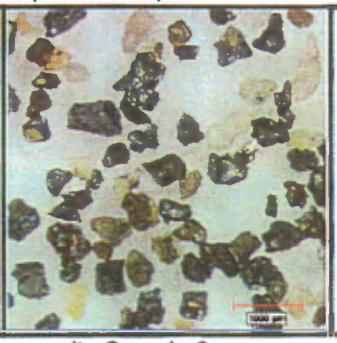
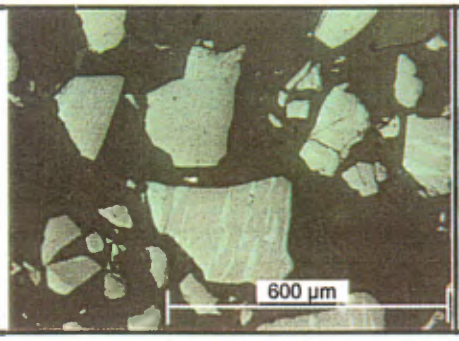
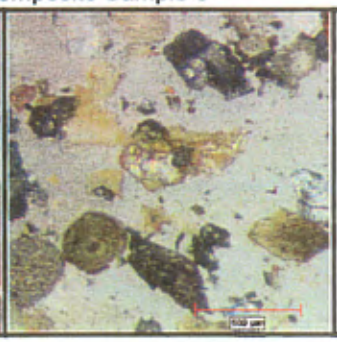
Composite Sample 2		
		**Particle size: +1180** The praticles are complex, they are composed of many minerals.
Composite Sample 3		
		**Particle size: 850-1180µm** No improvement in the liberation of minerals is evident. There are a number of minerals contained within each particle.

Table 4-8: Mineralogical Comparison of Composite Samples 4,5 and 6.

Composite Sample 4		
		**Particle size: 600-850µm** There seems to be a decrease in the amount of different mineral contained within the particles.
Composite Sample 5		
		**Particle Size: 300-600µm** There appears to be some liberation in this particle size range. The polished sections appear to have liberated ilmenite-hematite particles. The particle pictures show binary and ternary composite particles.
Composite Sample 6		
		**Particle Size: -300µm** There is a wide range of particle sizes in this sample, some minerals have been liberated while others are part of a composite particle.

4.3 CONCENTRATION

4.3.1 Heavy Liquid Separation

Heavy liquid separation was performed according to the methodology in section 3.3.3.A. Table 4-9 shows the division of material into sinks and floats. Composites 2 through 5 had a similar division of material between sinks and floats. Composite 6 showed a 10% increase in the amount of material going to sinks. There was a slight loss of material during this test.

Table 4-9: Heavy Liquids Separation Division of Material.

	Material	Sinks		Floats		Lost Material	
Sample ID	Initial wt (g)	wt (g)	% of Initial	wt (g)	% of Initial	wt (g)	% of Initial
Comp 2	55.63	44.55	80.08%	11.10	19.95%	-0.02	-0.04%
Comp 3	38.67	31.66	81.87%	6.96	18.00%	0.05	0.13%
Comp 4	49.34	40.43	81.94%	8.86	17.96%	0.05	0.10%
Comp 5	61.86	48.56	78.50%	12.95	20.93%	0.35	0.57%
Comp 6	62.18	43.06	69.25%	18.46	29.69%	0.66	1.06%

4.3.2 Magnetic Separation

Magnetic separation was conducted in accordance with the method outlined in sections 3.3.3.B. The ratio of magnetic to non-magnetic material generally increased as the particle size decreased (Table 4-10).

Table 4-10: Magnetic Separation Division of Material.

		Magnetics		Non-Magnetics		Lost Material	
Sample ID	Sink wt (g)	wt (g)	% of Sinks	wt (g)	% of Sinks	wt (g)	% of Sinks
Comp 2	44.55	33.81	75.89%	10.73	24.09%	0.01	0.02%
Comp 3	31.66	23.08	72.90%	8.57	27.07%	0.01	0.03%
Comp 4	40.43	34.10	84.34%	6.33	15.66%	0.00	0.00%
Comp 5	48.56	40.60	83.61%	7.87	16.21%	0.09	0.19%
Comp 6	43.06	37.06	86.07%	5.04	11.70%	0.96	2.23%

4.3.3 Assay Analysis of Separation Tests

The floats and sinks that were divided into magnetics and non-magnetics were analyzed by XRF. The XRF compositional analysis can be seen in Tables 4-11, 4-12 and 4-13. The elements that concentrated in the floats were silicon, aluminum, calcium, and sodium. These are components of silicate minerals. Titanium concentrated in the magnetics; this was the concentrate. The grade of TiO_2 in the concentrates was found by looking at the chemical analysis of the magnetic portion. The concentrate of comp 6 had a grade of 33.40% titanium oxide, this was the best result. The recovery of titanium was also the best with comp 6 at 97.70% (Figure 4-5). A mass balance of titanium dioxide for was calculated for the separation and concentration steps in Appendix B.

Table 4-11: Results of Assay by XRF of Floats.

	Composition %													
Sample ID	Fe2O3	TiO2	SiO2	Al2O3	MgO	CaO	Na2O	P2O5	V2O5	K2O	MnO	Cr2O3	LOI	Sum
Comp 2 Flt	5.87	1.9	51.4	24.1	1.42	8.35	4.82	0.34	0.02	0.73	0.02	0.01	0.49	99.5
Comp 3 Flt	5.36	1.66	51.2	24.4	1.33	8.48	4.78	0.3	0.03	0.72	0.02	0.01	0.49	98.8
Comp 4 Flt	4.8	1.42	52.4	25.1	1.18	8.66	4.85	0.27	0.02	0.77	0.02	0.02	0.57	100.1
Comp 5 Flt	4.13	0.96	51.7	25.3	1.02	9.13	4.82	0.62	0.01	0.77	0.01	0.01	0.8	99.3
Comp 6 Flt	6.72	1.28	41.9	20.6	1.19	15.1	3.69	6.49	0.03	0.57	0.02	< 0.01	1.88	99.4

Table 4-13: Results of Assay by XRF of Non-Magnetics.

	Composition %													
Sample ID	Fe2O3	TiO2	SiO2	Al2O3	MgO	CaO	Na2O	P2O5	V2O5	K2O	MnO	Cr2O3	LOI	Sum
Comp 2 Non Mag	42.7	21.3	22.6	2.35	10.3	1.65	0.32	0.68	0.22	0.05	0.23	0.02	-1.79	100.7
Comp 3 Non Mag	42.6	21.7	21.8	3.12	9.11	2.21	0.45	0.88	0.22	0.07	0.2	< 0.01	-1.75	100.6
Comp 4 Non Mag	29.4	8.87	39.2	2.61	17.9	1.72	0.13	0.68	0.1	0.05	0.27	< 0.01	-1.07	99.9
Comp 5 Non Mag	25.8	5.12	42.6	2.46	19.9	2.37	0.07	1.29	0.06	0.03	0.28	< 0.01	-0.39	99.6
Comp 6 Non Mag	23	1.04	36	2.64	16.4	9.46	0.15	6.75	< 0.01	0.03	0.22	< 0.01	2.96	98.7

Table 4-12: Results of Assay by XRF of Magnetics

	Composition %													
Sample ID	Fe2O3	TiO2	SiO2	Al2O3	MgO	CaO	Na2O	P2O5	V2O5	K2O	MnO	Cr2O3	LOI	Sum
Comp 2 Mag	51.4	28.1	12.4	1.99	5.94	1.27	0.18	0.56	0.28	0.06	0.18	< 0.01	-2.02	100.3
Comp 3 Mag	50.4	27.3	13.6	2.54	5.9	1.47	0.25	0.57	0.27	0.08	0.19	< 0.01	-1.92	100.6
Comp 4 Mag	52.6	29.5	11	1.54	5.7	1.05	0.13	0.48	0.29	0.03	0.19	< 0.01	-2.13	100.4
Comp 5 Mag	56.2	32	7.38	1.14	4.37	0.63	0.11	0.3	0.33	0.03	0.18	0.01	-2.21	100.5
Comp 6 Mag	61.5	33.4	3.09	0.91	2.5	0.42	< 0.05	0.23	0.35	0.02	0.17	0.02	-2.23	100.4

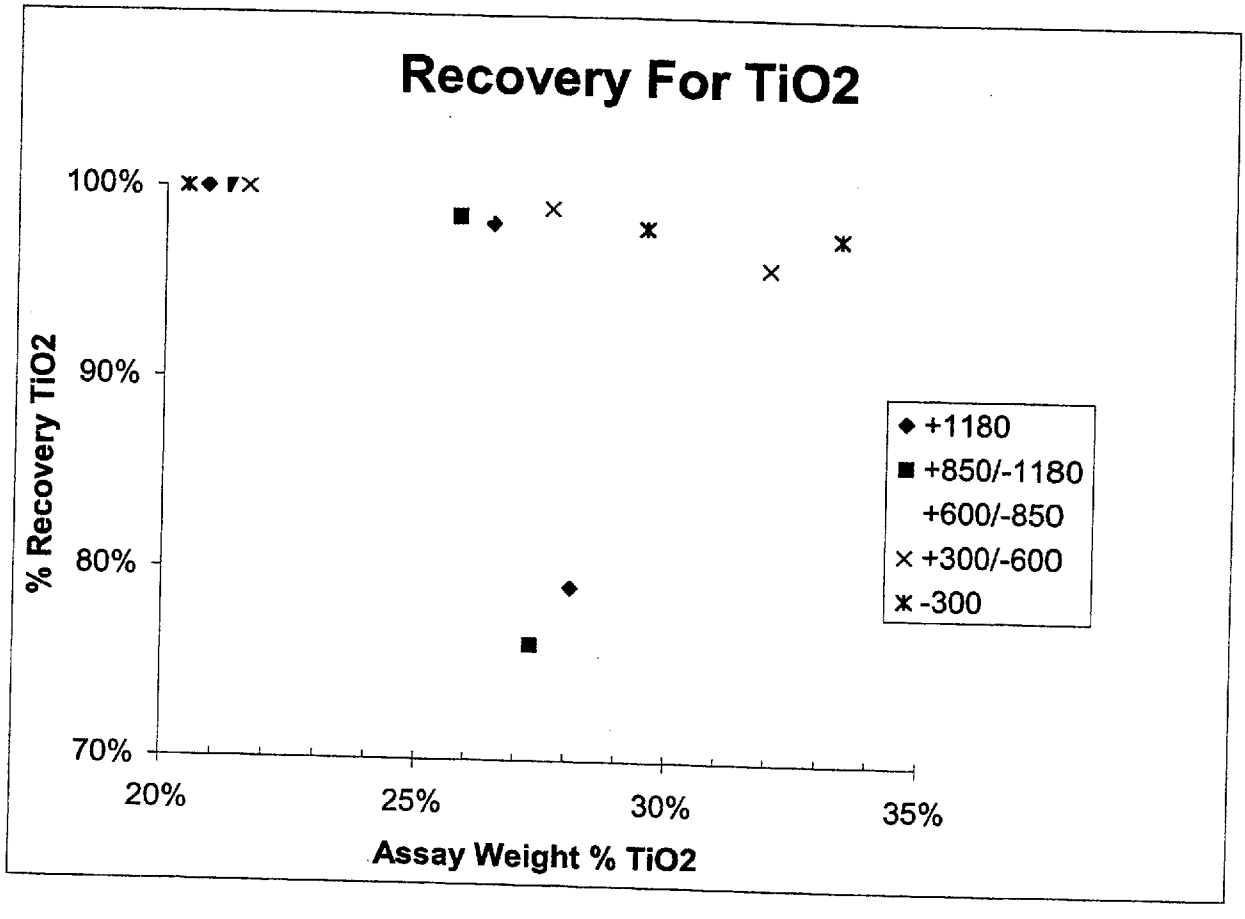


Figure 4-5: Recovery for Titanium in Floats, Non-Magnetics and Magnetic Fractions.

Chapter 5 DISCUSSION

The aim of this thesis was to make a concentrate of titanium with the best possible grade. In order to concentrate the titanium the following steps were taken: bulk compositional analysis, crushing and analysis, and separation.

The first thing that was done was a bulk sample compositional analysis. This was done to establish a starting point for the experiment. Prior to the concentration stage the crushed material was separated into several size fractions that were examined by XRF and by optical microscopy to note any differences between them.

In order to achieve the best concentrate grade possible several particle sizes were used for separation. The methods chosen for concentration were gravity separation, using heavy liquid separation, and magnetic separation.

5.1 Bulk Sample Analysis

The bulk sample was analyzed by chemical and mineralogical means. The chemical analysis by XRF found that 40% of the sample was Fe_2O_3 and 21.2% was TiO_2. Iron oxide was therefore present in twice the amount as titanium dioxide. Iron, was expected to be found since titanium is normally associated with an iron oxide of some kind. The chemical composition doesn't say much about the bulk sample, it simply lists the major compounds present. Mineralogical analysis was used to determine what type titanium and iron minerals were present and how they are associated. Since silica was also found to be a major constituent of the ore at 21.1%. This indicated that the material could be up graded by gravity separation through the removal of the lighter silicate minerals.

ICP was used to see if anything of value other than titanium and iron was in the sample. The trace element found the largest amount was Co at 0.023%. Copper, a valuable metal, was found to be 0.014% of the bulk sample. However, these amounts are very small thus not worth further exploration. Therefore the analysis by ICP revealed that there were no additional elements of value in the bulk of the samples.

Ore characterization found that the iron oxides present were hematite and magnetite. Both of these minerals were identified by XRD as being present in minor amounts. Optical microscopy on the polished sections showed that the hematite was intimately connected within the ilmenite. Fine needle like patterns could be found throughout the ilmenite grains. The close relationship between hematite and ilmenite indicated that it would not be practical to separate the two minerals through physical means. Therefore the particle liberation that was sought of the combined ilmenite-hematite particles from the surrounding material.

Ore characterization found that the surrounding material was composed mainly of feldspar and a few other minerals such as pyroxene, silicates, apatite, chalcopyrite and copper sulfide. These minerals were attached to the sides of the ilmenite grains, or sometimes inside, and in some cases had already been liberated. Therefore the ilmenite-hematite grains should be able to be liberated from the gangue through grinding.

5.2 Particle Size Analysis

Particle size classification resulted in the formation of 5 composite samples of different particle sizes. Chemical analysis by XRF found that the chemical composition was similar in each of the size fractions, therefore the sample had not segregated at any particle size. This indicated that the sample was homogenous.

Upon examination of the polished sections of the composite samples it was found that liberation of the ilmenite-hematite grains began at particle sizes between 600 and 850μm. The liberation increased with particle sizes between 300 and 600μm, and increased again when the particles were reduced in size to below 300μm. The grind size with the highest liberation was therefore comp 6 (-300μm).

5.3 Gravity Separation

Gravity separation using heavy liquids separated the lighter minerals from the heavier ones. The liquid used was methylene iodide with a density of 3.10g/cm^3. Those minerals with a density greater than 3.10g/cm^3 would sink and those with a smaller density would float. Silicates have a great diversity of chemical compositions and consequently have a wide range of density values ranging from less than 2g/cm^3 to

greater than 7.0g/cm^3. The majority of silicates have a density of around 3.0g/cm^3, thus they floated in the heavier methylene iodide. Ilmenite has a density between 4.5 and 5.0g/cm^3; this is average for metallic minerals. Magnetite has an average density of 5.1g/cm^3 and Hematite has a density of 5.3g/cm^3. All of these heavier minerals sank in the heavy liquid medium [4].

The heavy liquids separation test yielded similar splits between floats and sinks for the composite samples 2 through 5. The splits were around 80wt% sinks and 20% to floats. There was a dramatic increase in the amount of floats from comp 5 to comp 6, close to 10% greater. This suggests two things either more silicate minerals were liberated from the heavier minerals or the particle size was reduced to a point where it hindered the settling of the particles. There was no greater amount of titanium lost in the floats between comp 6 and the other composite samples.

5.4 Magnetic Separation

The sinks were put through a magnetic separator. The ilmenite-hematite minerals were attracted to the magnet and concentrated in the magnetic fraction. The magnetite is magnetic and would also concentrate with the magnetics. As the particle size decreased the magnetics portion increased. By reducing the particle size the amount of exposed magnetic material was increased; thus, attracting the particles to the magnet. There was little difference (0.6%) between the titanium oxide in the concentrates of comps 2 and 3. The next particle size down, comp 4, showed an increase in grade of 2.2%. The increase from comp 4 and 5 was 2.5%; and between comp 5 and 6, 1.4%. The best concentrate grade was therefore achieved by crushing the material to below 300μm. This resulted in an increased grade of titanium of 12.3% over the original bulk sample.

5.5 Limitations of this Experimental Work

The first limitation of the experiment had to do with the size of sample used. Five kilograms of material is not enough to draw conclusions from, nor is one set of data for each separation test.

Other limitations had to do with analytical techniques. XRF does not have the sensitivity necessary to detect lighter elements. Sample fusion enhances the XRF measurement technique by minimizing particle size effects but sometimes refractory minerals dissolve slowly and do not give satisfactory fusions. The other analytical technique employed was ICP. The emission spectra are complex and interelement interferences are possible if the wavelength of the element of interest is very close to that of another element. The sample must also be digested prior to analysis in order to dissolve the element(s) of interest, there is a significant amount of elements that are only partially digested and there is incomplete analysis.

Chapter 6 CONCLUSION AND FUTURE WORK

The goal of making a titanium concentrate with an increased grade over the original material was achieved. The best titanium concentrate grade was 33.40%. This was accomplished by gravity separation using heavy liquids and magnetic separation with a rare-earth magnetic separator. The maximum particle size used to attain this concentration grade was 300μm.

Future experimental directions to take in regards to concentration of this ore would be to examine the grind sizes of around 300μm more closely. Larger sample sizes should be used and separation tests should be repeated. Different magnetic strengths for magnetic separation could be investigated. Perhaps a more useful investigation would be to do an economical feasibility study to determine the minimum grade of titanium in the concentrate that could be sold. It would also be advantageous to know what the concentrate composition can contain, for example there may a detrimental element present that would reduce the value of the concentrate.

References

[1] Mineral Information Institute- Titanium http://www.mii.org/Minerals/phototitan.html

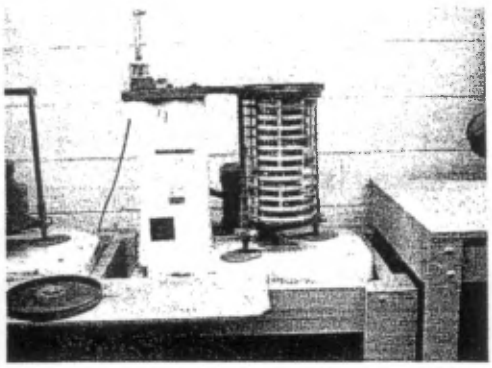
[2] http://www.azom.com/details.asp?ArticleID=1179

[3] Elliot R A, "Benefication of Titanium Ores With Particular Reference to Canadian Ores" The Canadian Mining and Metallurgical Bulletin for March, 1959, Montreal.

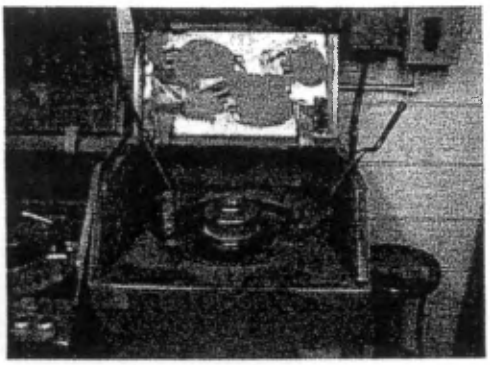
[4]Mineral Gallery: http://mineral.galleries.com

[5] http://www.chemex.com/tech/t-sec2_4.htm#sec2_4_1_3

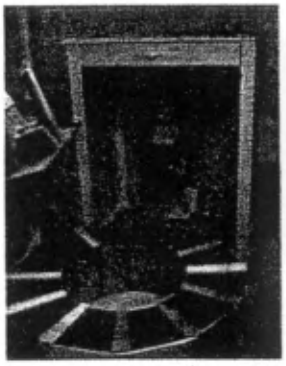
Jaw Crusher



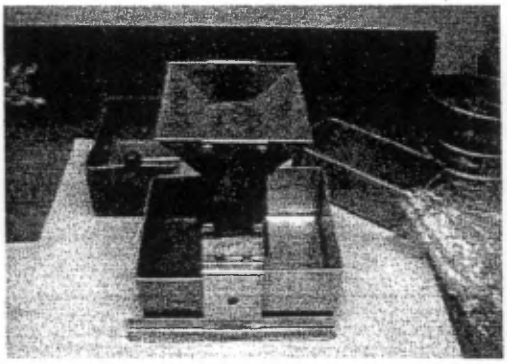
Ro-tap and Screens



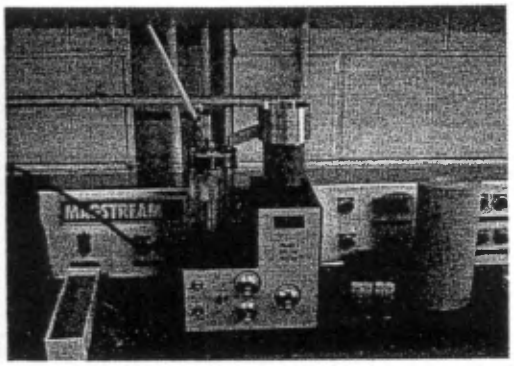
Pulverizer



Rotating Riffler



Riffler



Rotating Micro Riffler

Siemens D5000 Diffractometer

Figure 6-9: Claiss Fluxy

APPENDIX B MASS BALANCE OF TITANIUM DIOXIDE

TiO2 MASS BALANCE

Comp 2 +1180 microns

	Weight (g)	Weight %	Assay wt % TiO2	Weight TiO2	wt% Recovery TiO2
Magnetic	33.81	60.77%	28.10%	9.50	79.19%
Non-Magnetic	10.73	19.28%	21.30%	2.29	19.05%
Float	11.1	19.95%	1.90%	0.21	1.76%
Mags + Non-Mags	44.54	80.05%	26.46%	11.79	98.24%
Caculated Head	55.64	100.00%	21.56%	12.00	100.00%
Grade of Concentrate					**28.10%**

Comp 3 +850/-1180 microns

	Weight (g)	Weight %	Assay wt % TiO2	Weight TiO2	wt% Recovery TiO2
Magnetic	23.08	41.48%	27.30%	6.30	76.10%
Non-Magnetic	8.57	15.40%	21.70%	1.86	22.46%
Float	6.96	12.51%	1.66%	0.12	1.40%
Mags + Non-Mags	31.65	56.88%	25.78%	8.16	98.60%
Caculated Head	38.61	100.00%	21.44%	8.28	100.00%
Grade of Concentrate					**27.30%**

Comp 4 +600/-850 microns

	Weight (g)	Weight %	Assay wt % TiO2	Weight TiO2	wt% Recovery TiO2
Magnetic	34.10	61.29%	29.50%	10.06	93.58%
Non-Magnetic	6.33	11.38%	8.87%	0.56	6.78%
Float	8.86	15.92%	1.42%	0.13	1.52%
Mags + Non-Mags	40.43	72.66%	26.27%	10.62	98.83%
Caculated Head	49.29	100.00%	21.80%	10.75	100.35%
Grade of Concentrate					**29.50%**

Comp 5 +300/-600 microns

	Weight (g)	Weight %	Assay wt % TiO2	Weight TiO2	wt% Recovery TiO2
Magnetic	40.6	72.97%	32.00%	12.99	96.09%
Non-Magnetic	7.87	14.14%	5.12%	0.40	4.87%
Float	12.95	23.27%	0.96%	0.12	1.50%
Mags + Non-Mags	48.47	87.11%	27.64%	13.39	99.08%
Caculated Head	48.47	100.00%	27.89%	13.52	100.58%
Grade of Concentrate					**32.00%**

Comp 6 -300 microns

	Weight (g)	Weight %	Assay wt % TiO2	Weight TiO2	wt% Recovery TiO2
Magnetic	37.06	66.61%	33.40%	12.38	97.70%
Non-Magnetic	5.04	9.06%	1.04%	0.05	0.63%
Float	18.46	33.18%	1.28%	0.24	2.85%
Mags + Non-Mags	42.1	75.66%	29.53%	12.43	98.13%
Caculated Head	60.56	100.00%	20.92%	12.67	100.99%
Grade of Concentrate					**33.40%**

APPENDIX C CERTIFICATES OF ANALYSIS FOR XRF AND XRD CONDUCTED AT SGS LAKEFIELD RESEARCH LTD.

Lakefield Research
P.O. Box 4300 - 185 Concession St.
Lakefield - Ontario - KOL 2HO
Phone: 705-652-2038 FAX: 705-652-6441

Mineralogical Services LRL Canada
Attn : Nicki McKay/ Shauna Pedler

---, ---

Phone: ---
Fax:---

Thursday, February 20, 2003

Date Rec. : 18 February 2003
LR Report : **CA1803-FEB03**
Project : 8901-377
Client Ref : MI5004-Feb03

CERTIFICATE OF ANALYSIS

Sample ID	SiO2 %	Al2O3 %	Fe2O3 %	MgO %	CaO %	Na2O %	K2O %	TiO2 %	P2O5 %	MnO %	Cr2O3 %	V2O5 %	LOI %	Sum %
1: Comp 1	21.1	6.55	40.0	5.39	3.37	1.12	0.18	21.1	1.00	0.15	< 0.01	0.22	-0.90	99.4
2: Comp 2	23.0	7.02	39.2	5.69	3.05	1.23	0.22	20.8	0.60	0.15	0.01	0.22	-1.14	100.1
3: Comp 3	22.3	6.26	40.0	6.13	2.70	1.13	0.20	21.3	0.54	0.16	< 0.01	0.23	-1.26	99.7
4: Comp 4	22.2	5.80	40.2	6.58	2.48	0.96	0.18	21.4	0.48	0.17	0.02	0.24	-1.23	99.4
5: Comp 5	21.7	6.49	40.4	5.77	2.73	1.10	0.19	21.6	0.51	0.16	0.02	0.24	-1.06	99.9
6: Comp 6	17.9	7.07	40.7	3.26	5.67	1.09	0.20	20.4	2.74	0.12	< 0.01	0.24	-0.12	99.2

Ken Maley, B.Sc., C. Chem
Assistant Manager, Minerals

OnLine LIMS

University of Toronto
8901-377, MI5004-FEB03
Feb. 21/03

SGS Lakefield Research Limited
185 Concession St
Lakefield, ON K0L 2H0

Summary of Qualitative X-ray Diffraction Results

Sample	Crystalline Mineral Assemblage (relative proportions based on peak height)			
	Major	Moderate	Minor	Trace
Comp 1	ilmenite	plagioclase	hematite, magnetite pyroxene	

*Tentative identification due to low concentrations, diffraction line overlap or poor crystallinity

Instrument: Siemens D5000 diffractometer
Scan Conditions: Co radiation, graphite monochromator, 40Kv, 30mA, Step: 0.02°, Step time:1s
Interpretations: JCPDS / ICDD powder diffraction files. Siemens Search / Match software.
Detection Limit: 0.5-2%. Strongly dependent on crystallinity.
Interpretations do not reflect the presence of non-crystalline / amorphous compounds. Mineral proportions are based on relative peak heights and may be strongly influenced by crystallinity, structural group or preferred orientations. Interpretations and relative proportions should be accompanied by supporting petrographic and geochemical data (WRA, ICP-OES).

Mineral	Composition
Hematite	Fe_2O_3
Ilmenite	$FeTiO_3$
Magnetite	Fe_3O_4
Plagioclase	$(NaSi,CaAl)AlSi_2O_8$
Pyroxene	$(Ca,Na)(Mg,Fe,Al,Ti)(Si,Al)_2O_6$

Note: (N/A)

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The Qualitative XRD method (METH # 8-8-1) used by SGS Lakefield Research Limited Mineralogical Services is accredited to the IEC/ ISO Guide 25 standard by the Standards Council of Canada. Mini-method available upon request.

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February 21, 2003

Date Rec. : 20 February 2003
LR Report : CA1805-FEB03
Project : 8901-377

CERTIFICATE OF ANALYSIS

Lakefield Research Limited - Final Report

Sample ID	SiO2 %	Al2O3 %	Fe2O3 %	MgO %	CaO %	Na2O %	K2O %
1: Comp 1 Flt	52.2	25.3	4.66	1.02	8.78	4.95	0.74
2: Comp 1 Mag	9.31	1.67	54.4	4.77	1.00	0.10	0.05
3: Comp 1 Non Mag	30.1	2.75	32.2	13.3	5.58	0.26	0.07
4: Comp 2 Flt	51.4	24.1	5.87	1.42	8.35	4.82	0.73
5: Comp 2 Mag	22.6	2.35	42.7	10.3	1.65	0.32	0.05
6: Comp 2 Non Mag	12.4	1.99	51.4	5.94	1.27	0.18	0.06
7: Comp 3 Flt	51.2	24.4	5.36	1.33	8.48	4.78	0.72
8: Comp 3 Mag	13.6	2.54	50.4	5.90	1.47	0.25	0.08
9: Comp 3 Non Mag	21.8	3.12	42.6	9.11	2.21	0.45	0.07
10: Comp 4 Flt	52.4	25.1	4.80	1.18	8.66	4.85	0.77
11: Comp 4 Mag	11.0	1.54	52.6	5.70	1.05	0.13	0.03
12: Comp 4 Non Mag	39.2	2.61	29.4	17.9	1.72	0.13	0.05

Sample ID	TiO2 %	P2O5 %	MnO %	Cr2O3 %	V2O5 %	LOI %	Sum %
1: Comp 1 Flt	1.20	0.38	0.01	0.01	< 0.01	0.89	100.2
2: Comp 1 Mag	30.0	0.45	0.18	< 0.01	0.31	-2.09	100.2
3: Comp 1 Non Mag	12.4	3.69	0.21	< 0.01	0.14	-0.58	100.1
4: Comp 2 Flt	1.90	0.34	0.02	0.01	0.02	0.49	99.5
5: Comp 2 Mag	21.3	0.68	0.23	0.02	0.22	-1.79	100.7
6: Comp 2 Non Mag	28.1	0.56	0.18	< 0.01	0.28	-2.02	100.3
7: Comp 3 Flt	1.66	0.30	0.02	0.01	0.03	0.49	98.8
8: Comp 3 Mag	27.3	0.57	0.19	< 0.01	0.27	-1.92	100.6
9: Comp 3 Non Mag	21.7	0.88	0.20	< 0.01	0.22	-1.75	100.6
10: Comp 4 Flt	1.42	0.27	0.02	0.02	0.02	0.57	100.1
11: Comp 4 Mag	29.5	0.48	0.19	< 0.01	0.29	-2.13	100.4
12: Comp 4 Non Mag	8.87	0.68	0.27	< 0.01	0.10	-1.07	99.9

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LR Report : **CA1805-FEB03**

Sample ID	SiO2 %	Al2O3 %	Fe2O3 %	MgO %	CaO %	Na2O %	K2O %
13: Comp 5 Flt	51.7	25.3	4.13	1.02	9.13	4.82	0.77
14: Comp 5 Mag	7.38	1.14	56.2	4.37	0.63	0.11	0.03
15: Comp 5 Non Mag	42.6	2.46	25.8	19.9	2.37	0.07	0.03
16: Comp 6 Flt	41.9	20.6	6.72	1.19	15.1	3.69	0.57
17: Comp 6 Mag	3.09	0.91	61.5	2.50	0.42	< 0.05	0.02
18: Comp 6 Non Mag	36.0	2.64	23.0	16.4	9.46	0.15	0.03
19-DUP: Comp 4 Flt	51.8	24.8	4.78	1.19	8.57	4.87	0.77

Sample ID	TiO2 %	P2O5 %	MnO %	Cr2O3 %	V2O5 %	LOI %	Sum %
13: Comp 5 Flt	0.96	0.62	0.01	0.01	0.01	0.80	99.3
14: Comp 5 Mag	32.0	0.30	0.18	0.01	0.33	-2.21	100.5
15: Comp 5 Non Mag	5.12	1.29	0.28	< 0.01	0.06	-0.39	99.6
16: Comp 6 Flt	1.28	6.49	0.02	< 0.01	0.03	1.88	99.4
17: Comp 6 Mag	33.4	0.23	0.17	0.02	0.35	-2.23	100.4
18: Comp 6 Non Mag	1.04	6.75	0.22	< 0.01	< 0.01	2.96	98.7
19-DUP: Comp 4 Flt	1.39	0.28	0.02	< 0.01	< 0.01	0.57	99.1

Nicole Mozola, B.Sc. (Eng)
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Mineralogical Characterization and Concentration of Everett Deposit Ilmenite Ore

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Abstract

Ilmenite from the Everett Deposit in Quebec has been characterized both chemically and mineralogically. The sample investigated contained 40% Fe_2O_3, 21% TiO_2 and 21% SiO_2 as the main components. The ilmenite grains contain about 25% hematite intergrown blades with widths ranging from 5 to 30 μm.

Crushing the sample to 0.6 mm or less lead to significant separation of the ilmenite from the gangue minerals. Gravity separation followed by magnetic separation for these sizes, lead to a TiO_2 concentrate of 30% TiO_2 or above at a recovery of more than 94%.

Background

Titanium has many uses both as metallic titanium and titanium dioxide. About 90% of the titanium market is pigment grade TiO_2(titania) which, among other applications, is used in whitening paints, plastics, paper and rubber. Metallic titanium is a highly desirable metal since it is hard, has a high melting temperature, is lightweight and corrosion resistant. These properties make titanium and its alloys very useful in the aerospace industry where materials need to withstand extreme temperatures and have a high strength to mass ratio. The aerospace industry accounts for 60% of the metallic titanium market. Because of the cost of producing titanium, its uses have so far mainly been limited to special applications. However, recently there have been new developments in the production process of metallic titanium(1-3), possibly leading to increased production and lower cost of titanium. Due to these developments possibly leading to increased demands of TiO_2, this project was initiated.

Canada has several large deposits of ilmenite($FeTiO_3$) in Quebec. The Quebec Iron and Titanium Corporation (QIT) has been mining one of these deposits(the Allard Lake orebody) since the 1950's(4, 5). Situated in the vicinity of the Allard Lake orebody, there is another large but lower grade ilmenite deposit(Everett titanium deposit)(6). The objective of this investigation was to characterize a sample from this deposit and determine if simple upgrading techniques could be use to separate the ilmenite from the gangue material.

Allard lake ilmenites contain blades or llameallae of hematite(4, 5). The ores have exsolved hematite lamellae in the host ilmenite. The hematite lameallae in many cases traverses the whole grain of ilmenite and may range in width from 0.1 mm down to small needles. This intimate relationship between the minerals, make it very difficult to separate the two by mechanical means. Gangue mineral associated with this ore are

mainly plagioclase feldspars and in smaller proportions apatite, hypersothene (a magnesium iron silicate), and mica(5). Test-work on the ore mined by the QIT Corporation showed that liberation of ilmenite-hematite from gangue is almost complete at 0.635 mm. Methods investigated for concentration of such ores include a) flotation, b) gravity separation, c) electrostatic separation and d) high-intensity magnetic separation.

Experimental

To prepare samples for chemical, mineralogical and size analysis a 5 kg sample from the Everett titanium deposit was crushed to less than 1.7 mm in a jaw crusher. One quarter of the crushed sample was kept for chemical and mineralogical analysis while the rest was sent to a Ro-tap for size classification with mesh sizes 12, 16, 20, 30, 40, 50, 70, 100, 150, 200, 270, 400. Each fraction was weighed to find the particle size distribution. The 13 fractions were then combined to make 5 classes of particle sizes of approximately the same weight. The samples were labeled composite S1 through S5 where composite S1 was composed of the largest particle sizes and S5 was the smallest. XRD, XRF, and ICP analysis required the sample to be homogenous and have a fine particles size between 5 and 10μm. The necessary particle size was achieved by grinding the sample in a pulverizer for around ~40 sec.

Chemical analysis of the major components was achieved by XRF(Bruker 3400 XRF spectrometer) which involved mixing 0.5 g of powered sample with 5.0 g of lithium metaborate into a platinum crucible, followed by the addition of 1 ml of ammonium nitrate. The ammonium nitrate was used as an oxidant to ensure complete oxidation of the elements. The crucible was fused for 2 minutes, poured into a disk and cooled quickly. This made a solid homogeneous glass disk approximately 3 cm in diameter that was placed in the XRF spectrometer. Loss on ignition (LOI) was found by placing one gram of each powdered sample in a furnace at a temperature of 1010 °C for 1 hour. The sample was then weighed to calculate the amount of material lost on ignition.

ICP(Radial Varian Vista ICP spectrophotometer) analysis of minor elements required approximately 1 g of the powdered sample. The sample underwent a 4 acid digestion with hydrochloric, nitric, hydrofluoric and perchloric acids, and brought up in a final matrix of 20% hydrochloric acid. The sample solution was then placed into the core of an inductively-coupled plasma at a temperature of approximately 8000 °C.

The major minerals present were determined by XRD(Siemens D5000 diffractometer) on 2 g of the powdered sample. The scan conditions were Co radiation, graphite monochromator, 40 kV, 30 mA, steps of 0.02° with a step time of 1 second. The detection limit was 0.5-2%. Optical microscopy was performed to identify minerals below the detection limit of the diffractometer and to determine the associations between the minerals. Thin polished sections were examined by transmitted light and the polished sections were examined with reflected light. Samples for optical microscopy were placed in a petri dish and examined for mineral associations with light microscopy. Other samples were made into thin polished sections using a 2-part cold setting epoxy resin.

Heavy liquid separation was performed using methylene iodide which has a density of 3.1 g/cm^3. This was done to separate the heavier minerals such as ilmenite from the lighter silicates. The material was therefore divided into two portions; floats for light

minerals(mainly silicates) and sinks for heavy minerals. After separation, all samples were washed in acetone and subsequently dried in an oven. The sinks were then put through a magnetic separator(rare-earth) of Gauss strength ~1200, separating it into a magnetic and a non-magnetic section. The non-magnetic material was passed through the magnetic separator for a second time to ensure that all magnetics were collected.

Results

A representative fraction of the overall sample was analyzed to establish a starting point for the experiment. The chemical composition of the head sample is given in Table 1 and Figure 1 for the major oxides and in Table 2 for the minor constituents(in ppm).

Table 1. Composition of ilmenite sample(mass%)

Fe_2O_3	TiO_2	SiO_2	Al_2O_3	MgO	CaO	Na_2O	P_2O_5	V_2O_5	K_2O	MnO
40.0	21.1	21.1	6.55	5.39	3.37	1.12	1.0	0.22	0.18	0.15

The sample contains 40% Fe_2O_3 and 21.1% TiO_2, corresponding to an iron to TiO_2 ratio of 1.37. This value is similar to that reported by Richard(6) but slightly higher than that of the Allard deposit(Fe/TiO_2 = 1.18) which has a TiO_2 content of about 35% and a total iron content of 40%. Silica was also found to be a major constituent of the ore at 21.1%. This indicates that the material could be up-graded by gravity separation through the removal of the lighter silicate minerals.

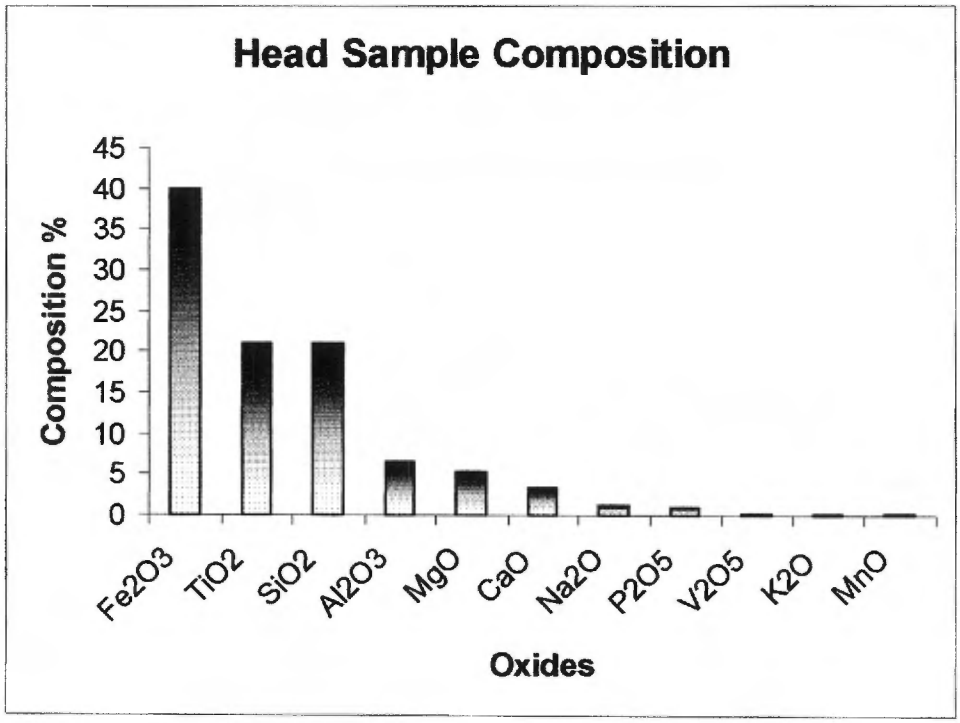

Figure 1. Chemical Composition of Ilmenite Sample

Table 2 shows that there are only minor amounts of other valuable metals such as Ag, Co, Cu and Ni. This implies that only TiO_2 and potentially iron, are of any economic value.

Table 2. Minor element content in ppm(or gram/tonne)

Ag	As	Ba	Be	Bi	Cd	Co	Cr	Cu	Li	Mo
< 2	< 50	78	< 1	< 30	< 5	230	62	140	< 25	26
Ni	**Pb**	**Sb**	**Se**	**Sn**	**Sr**	**Tl**	**U**	V	Y	Zn
88	63	< 20	< 100	< 100	200	< 100	< 75	940	4.3	64

Ore characterization using XRD revealed that ilmenite was the major mineral present, then plagioclase(feldspar), followed by hematite, magnetite, and pyroxene. Optical microscopy also confirmed the presence of these minerals. Several other minerals were also identified including pyrite, chalcopyrite, mica, muscovite, and quartz, goethite and rutile, and apatite (a phosphate). These minerals were attached to the sides of the ilmenite grains, or sometimes inside, and in some cases had already been liberated. Therefore it should be possible to liberate the ilmenite-hematite grains from the gangue through grinding.

Optical microscopical images in Fig. 2 show that the hematite was intimately connected within the ilmenite. Fine needle like patterns and bands are found throughout the ilmenite grains. The blades range from about 5 μm to about 30 μm in width, sometimes traversing through the whole ilmenite grains. The area fraction of hematite in the ilmenite grains varies approximately from 20 to 25%, leading to a hematite mass fraction of about 25%. This close interlocking between hematite and ilmenite indicates that it is not practical to separate them through physical means. The highest grade of TiO_2 in any concentrate formed by physical separation means is therefore limited to about 40%.

Using the Ro-tap and screens from +12 mesh to –400 mesh, the particle size distribution was determined. The results are given in Table 3 and illustrated in Fig. 3. The size fractions were then consolidated into 5 composite samples of approximately equal weight (Table 3). These samples were then used in the subsequent gravity and magnetic separation tests. The purpose was to determine what size reduction is required in order to allow for sufficient separation of the ilmenite grains from the gangue materials.

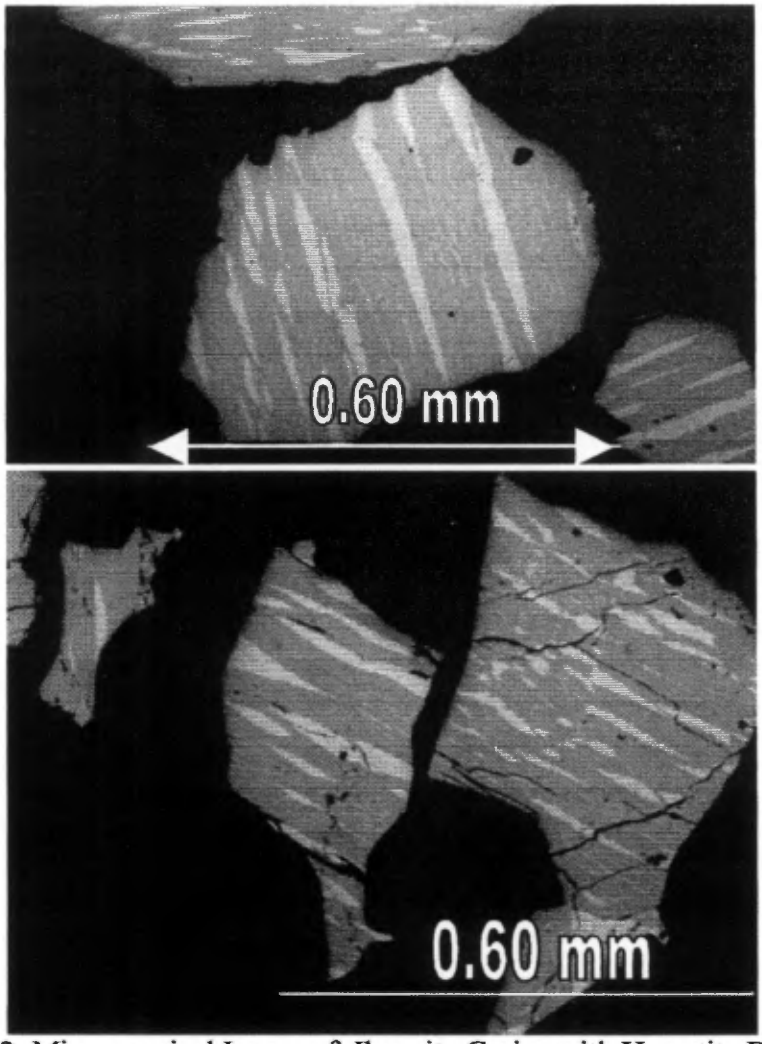

Figure 2. Microscopical Image of Ilmenite Grains with Hematite Blades

Table 3. Screen analysis of bulk sample.

Sample	Mesh	Microns	Weight %	Cumulative	Weight %
Size 1	12	1700	0.5	100.0	20.1
	16	1180	19.6	99.5	
Size 2	20	850	17.9	79.9	17.8
Size 3	30	600	17.2	62.1	17.2
Size 4	40	425	13.1	44.9	22.6
	50	300	9.5	31.8	
Size 5	70	212	7.9	22.3	22.3
	100	150	4.7	14.3	
	150	106	3.7	9.6	
	200	75	2.2	5.9	
	270	53	1.4	3.8	
	400	38	0.9	2.4	
	pan	-38	1.5	1.5	

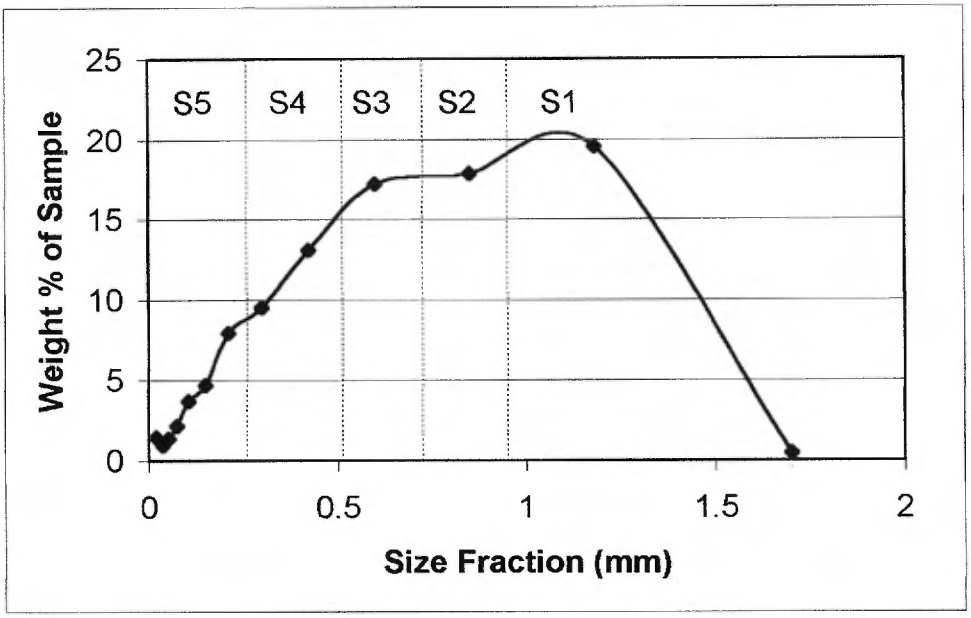

Figure 3. Size distribution of bulk sample after being passed through the jaw crusher. The division of the five size ranges used in further separation testing is also indicated.

XRF analysis of the five different size fractions showed that the composition was nearly identical for all sizes. This indicates that the sample was homogenous and that very little segregation has taken place during this sample preparation stage. The exception was the finest size where there was a 3% drop in the SiO_2 content with some increase in the CaO and P_2O_5 contents. The TiO_2 and Fe_2O_3 contents stayed nearly constant with a standard deviation of less than 0.5% between the various size fractions.

Gravity separation using a heavy liquid(3.10 g/cm^3) separated the lighter minerals from the heavier ones. Silicates generally have a density of less than 3.0g/cm^3 and would therefore float, while ilmenite, magnetite and hematite have densities from about 4.5 to 5.4 g/cm^3 and would therefore sink. Heavy liquid separation was performed on the five size fractions given in Table 3. The results shown in Table 4 give splits of around 80 wt% sinks and 20% to floats for sample sizes 1 through 4. Probably due to improved particle separation, the smallest sample(size 5) showed a 10% increase in the amount of material going to floats.

Table 4: Heavy Liquid Separation Division of Material.

Sample ID	Initial weight (g)	Sinks % of Initial	Float % of Initial	Lost sample
Size 1	55.63	80.1%	20.0%	-0.04%
Size 2	38.67	81.9%	18.0%	0.13%
Size 3	49.34	81.9%	18.0%	0.10%
Size 4	61.86	78.5%	20.9%	0.57%
Size 5	62.18	69.3%	29.7%	1.06%

The sinks were subsequently put through a magnetic separator to separate the non-magnetite gangue from the ilmenite concentrate. As the particle size decreased the magnetic portion increased. By reducing the particle size, the amount of exposed magnetic material was increased; thus, attracting the particles to the magnet. There was little difference (0.6%) between the titanium oxide in the concentrates of sizes 1 and 2. The next particle size down, size 3, showed an increase in grade of 2.2%. The increase from size 3 to 4 was another 2.5%, and down to the smallest size another 1.4%. The best concentrate grade was therefore achieved by crushing the material to below 300μm. This resulted in an increased grade of titanium of 12.3% over and above the original bulk sample. Figure 4 and Table 5 give the distribution of the major oxides.

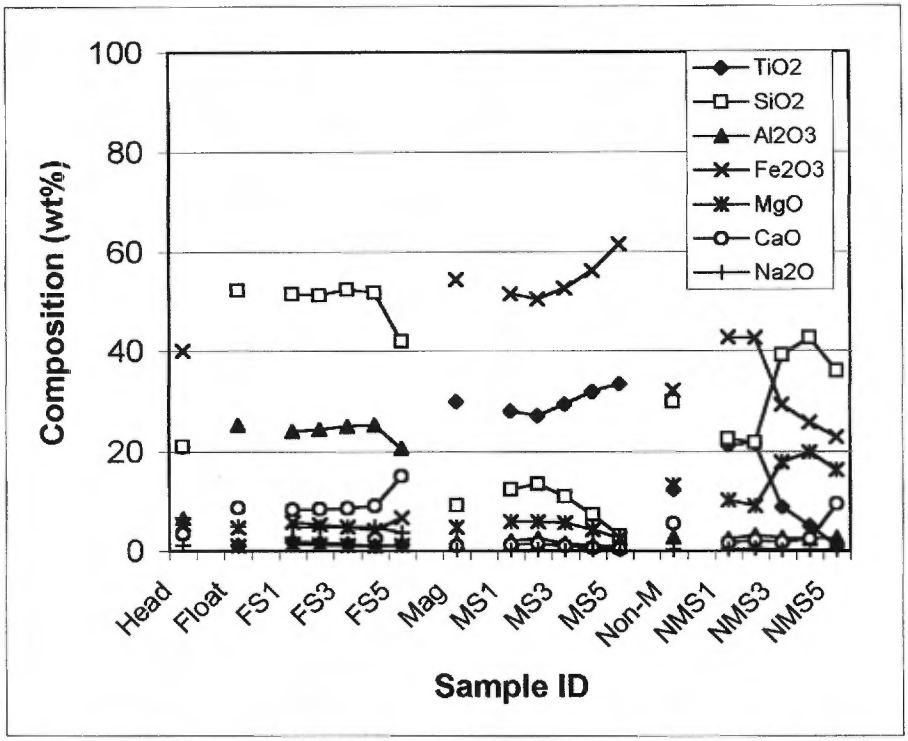

Figure 4 shows the composition of the major oxides varied between the various size fractions for the a) float, b) magnetic and c) non-magnetitc fractions.

Table 5. Composition of various size fractions of float, magnetic and non-magnetic fractions. The %mass recovered and the $\%TiO_2$ recovery in the float versus magnetic versus non-magnetic fractions for the various size fractions are also given.

	TiO_2	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	% Mass	$\%TiO_2$ recovery
Comp Float	1.2	52.2	25.3	4.66	1.02	8.78	4.95	20.3	1.2
F-S1	1.9	51.4	24.1	5.87	1.42	8.35	4.82	20.0	1.8
F-S2	1.66	51.2	24.4	5.36	1.33	8.48	4.78	18.0	1.4
F-S3	1.42	52.4	25.1	4.8	1.18	8.66	4.85	18.0	1.2
F-S4	0.96	51.7	25.3	4.13	1.02	9.13	4.82	21.0	0.9
F-S5	1.28	41.9	20.6	6.72	1.19	15.1	3.69	30.5	1.9
Comp Mag	30	9.31	1.67	54.4	4.77	1.0	0.1	63.9	90.7
M-S1	28.1	12.4	1.99	51.4	5.94	1.27	0.18	60.8	79.2
M-S2	27.3	13.6	2.54	50.4	5.9	1.47	0.25	59.8	76.1
M-S3	29.5	11	1.54	52.6	5.7	1.05	0.13	69.2	93.6
M-S4	32	7.38	1.14	56.2	4.37	0.63	0.11	66.1	96.1
M-S5	33.4	3.09	0.91	61.5	2.5	0.42	<0.05	61.2	97.7
Comp Non-M	12.4	30.1	2.75	32.2	13.3	5.58	0.26	15.9	9.3
NM-S1	21.3	22.6	2.35	42.7	10.3	1.65	0.32	20.0	19.1
NM-S2	21.7	21.8	3.12	42.6	9.11	2.21	0.45	22.2	22.5
NM-S3	8.87	39.2	2.61	29.4	17.9	1.72	0.13	12.8	5.2
NM-S4	5.12	42.6	2.46	25.8	19.9	2.37	0.07	12.8	3.0
NM-S5	1.04	36	2.64	23	16.4	9.46	0.15	8.3	0.4

The ratio of magnetic to non-magnetic material generally increases as the particle size decreases. The elements that concentrated in the floats were silicon, aluminum, calcium, and sodium. TiO_2 concentrated in the magnetic fraction that also would be the final product from this upgrading process. It is seen that for sizes 1 and 2 corresponding to particles larger than 850 μm the TiO_2 content in the magnetic section was noticeably lower than for the finer screen sizes. This is also supported by microscopic examination where it was found that liberation of the ilmenite-hematite grains began at particle sizes below about 850 μm. The liberation increased with particle sizes between 300 and 600μm, and increased again when the particles were reduced in size to below 300μm. The grind size with the highest liberation was therefore size 5 (-300μm).

The titania to silica ratios in Fig.5 show that as the screen size decreases, the relative amount of TiO_2 to SiO_2 in the magnetic fraction increases. This means that with finer crushing it is possible to separate the silica gangue from the ilmenite. For the TiO_2 to Fe_2O_3 ratio there is no strong dependency on the size of the sample on the magnetics. However, for the non-magnetics the finer the sample size the more Fe_2O_3 is removed relative to TiO_2.

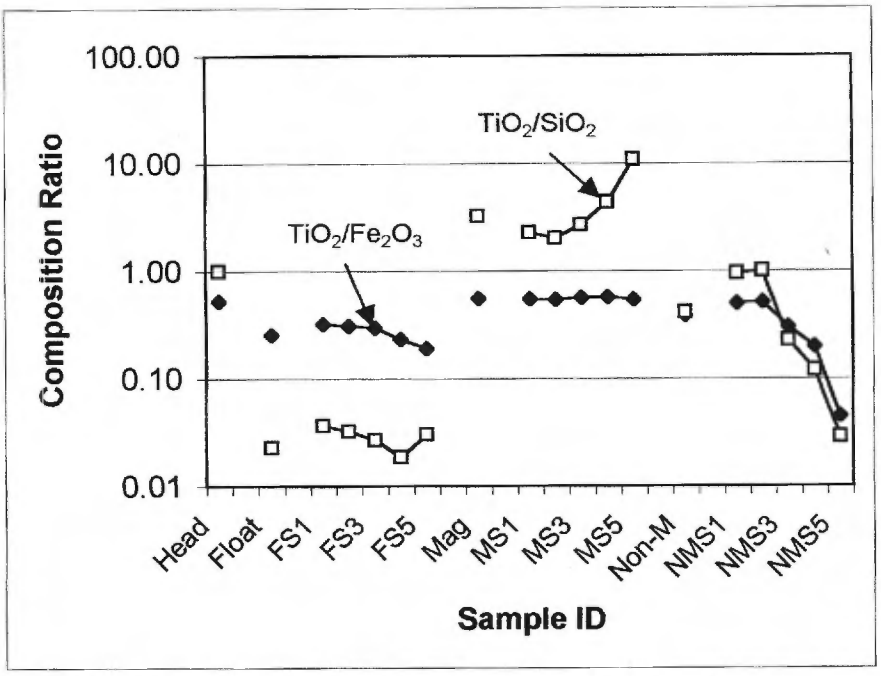

Figure 5. TiO_2 to Fe_2O_3 and TiO_2 to SiO_2 and ratios as a function screen size and sample location

In terms of TiO_2 recoveries for the various sizes, it is seen that for grains equal or less than 0.6 mm, the recovery to the magnetics is greater than 94%. In addition, these sizes also have the highest TiO_2 grade of 30% or more. Very little TiO_2 is lost in the float indicating that ilmenite is not tightly associated with light silica based minerals.

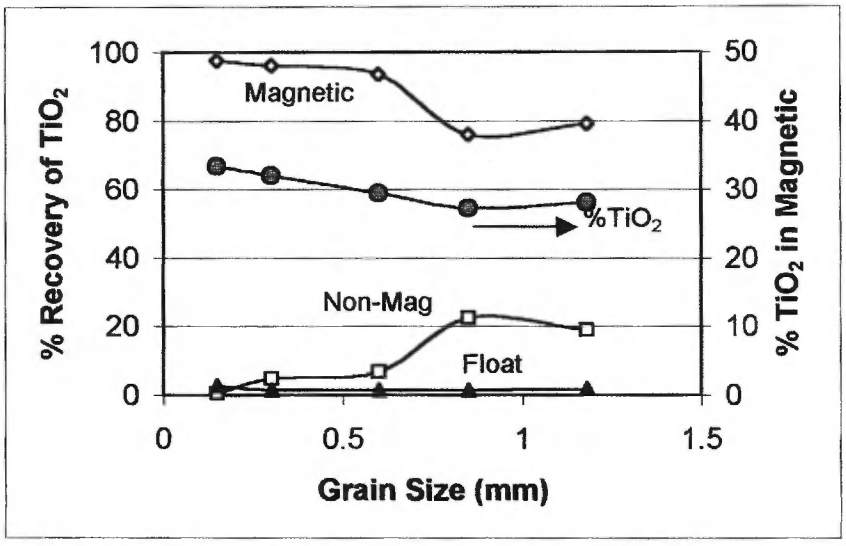

Figure 6. Recovery of TiO_2 in the various samples as a function of grain size. Also given is the $\%TiO_2$ in the magnetic fraction.

Conclusions

Everett ilmenite samples contain 5 to 30 μm wide lamella of hematite intergrown within the ilmentite grains. The grains contain typically about 75% ilmenite and 25% hematite, with occasionally the presence of other gangue minerals. Using a head sample with 21% TiO_2, gravity and magnetic separation produced a concentrate of above 30% TiO_2. The highest grade of 33.4% TiO_2 was achieved with samples crushed to less than 0.3 mm. The recovery of TiO_2 in samples crushed to less than about 0.65 mm was higher than 95%.

Future experimental with larger sample sizes should be carried out. Tests should also be carried out to determine if indeed it is necessary to carry out gravity separation before magnetic separation. Based on improved liberation at samples sizes from about 0.6 mm and down, further tests should be carried out to determine the actual required size reduction necessary. Different magnetic strengths for magnetic separation could be investigated.

Acknowledgement. The technical support and expertise provided by SGS Lakefield Research Ltd. and the financial support from NSERC are greatly appreciated.

Natural Science + Engineering Research Council

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