Pelletization and Direct Reduction of Local Iron Oxide

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Abstract

Methods of pelletization and direct reduction with local iron oxide concentrates were explored. The strength of the pellets was tested and the strong pellets were tested for reduction speeds in a quartz tube furnace. Two types of pellets were tested, carbon infused pellets and non-carbon pellets. The results of the tests were inconclusive, but they provided a basis for future work to be done.

Introduction

This NSF project is another step in enabling the students to be able to create iron and eventually steel which will be used to manufacture a katana, Samurai sword. The previous year was the initiation of the Samurai sword project, and ended with disappointing results. An overambitious furnace design coupled with very little material testing resulted in only partially purified pellets. From these ashes arose this summer's work. In an attempt to gain a better grasp on the kinetics of reducing iron, a series of test have been conducted to decide on a starting material and how long this selected material will need to reduce under different atmospheres and pellet composition.

The objective of this work is to determine a way to reduce native iron ore by direct reduction in a primitive blast furnace. Research will be conducted on what native iron ore will be best for pelletization and reduction into iron. The goal of the pelitization process is to produce pellets of approximately 4-6 mm in diameter that would be strong enough to be put into a blast furnace. Research will then be conducted on making strong pellets that reduce quickly.

Background Information

In order to create iron out of an iron oxide feed material the material has to be reduced fully to elemental iron before it is melted. If melting occurs before the iron is fully reduced the liquid phase will become slag, a solution of molten iron oxides that is a typical byproduct of steelmaking. This summer was devoted to further this knowledge to enable next year's metallurgical design teams to produce a fully iron mass that can be converted into steel. A base knowledge of direct iron reduction is needed to be able to reduce the iron oxides efficiently.

The thermodynamics of iron reduction can be seen from the standpoint of an Ellingham diagram provided in Figure 1. Notice the hematite (Fe_2O_3) to magnetite (Fe_3O_4) reaction is at the top, thus this oxide has the highest oxidizing potential. Following down the diagram are the magnetite to wustite and magnetite to iron reactions. All of these reactions fall in the top region of the diagram which demonstrates that the oxidizing potential for them is quite high and readily occur when above 1000°C where it becomes preferential for CO to exist in the atmosphere than as CO_2. Weight percentage ratios of iron to oxygen also follow this order of reduction. Hematite is 69.94 wt% Fe and as it changes to magnetite becomes 72.36 %wt until you reach iron.
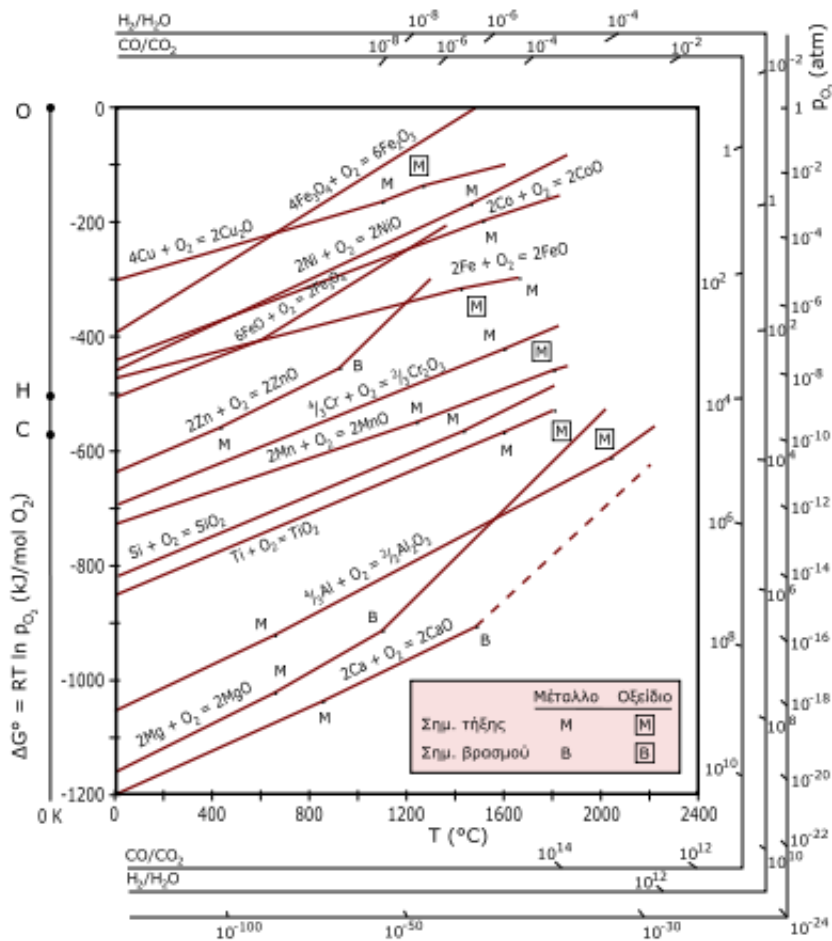

Figure 1: Ellingham Diagram

In direct iron reduction the feed material is pellets that are largely composed of hematite. The porosity and size of the pellets as well as the atmosphere of the furnace essentially determine the reduction rate of iron pellets. This process works in three modes, diffusion of carbon with oxygen to create CO, the oxidation of CO gases to CO_2 gases, and the formation of water vapor H_2O from free hydrogen and oxygen within the ore. The largest contributor to this process is by far the reaction of CO to CO_2. Direct reduction processes as well as blast furnaces obtain the carbon for these reactions from coke sources that are poured in layers with the feed material.

As mentioned previously, porosity is by far the most influential factor in the reducibility of pellets. The greater porosity in a pellet the more surface area is exposed to the atmosphere, thus the faster the reactions are able to occur. Crystal structure of the oxides also plays a role in this. Hematite has a crystal structure that is hexagonal close-packed, however magnetite and wustite are face centered cubic and this change in crystal structure creates a 25% increase in volume (1). The affect of this factor can be most easily seen when considering the three kinetic resistances involved with reducing iron pellets with the carbon monoxide molecule. The first is the diffusion of the CO in the pellet to its reaction point, next is the reaction from CO to CO_2, and finally the diffusion of the CO_2 gas out of the pellet. In order to simplify the kinetics of our processes we have chosen to keep the pellets relatively small in order to mitigate these factors. Keeping the pellets small reduces the distance the carbon monoxide has to diffuse as well as increasing the surface area of the bed of pellets.

Broader Impact

This summer's research has shed some light on the complexity of iron reduction and making steel. Students have realized the difficulties of the process and how amazing the feat of producing steel is without any modern technology. The Japanese produced steel by using a iron-bearing river sand. A tatara, a large rectangular furnace made from clay, was used for the iron reduction and steelmaking. One batch of steel would take about 25 tons of sand and charcoal with the tatara operating at a temperature of about 2500 degrees F for about 3 days. To control the amount of carbon dissolved into the steel, molten temperatures were never reached throughout the process. Japanese men were able to determine the amount of carbon content based solely on the way the steel broke apart.

Japanese swordsmiths used two different types of steel in the making of a katana, high and low carbon steel. The high carbon steel was shaped into a long U-shaped channel, and the low carbon steel was shaped to fit and then placed inside the high carbon steel. This is done to take advantage of the different properties of the two types of steel. The low carbon core gives the sword its toughness and allows for shock absorption. The high carbon outer shell allows the sword to maintain a razor-sharp edge.

After the blade has been forged, the swordsmith will coat the blade with a clay and charcoal powder mixture. Thickness of the mixture will be varied on the blade with the dull edge having the thickest layer and the sharp edge having the thinnest. The blade will then be reheated to a temperature of about 1500 degrees F. This process gives the blade a wavy design known as the hamon. To produce the curvature that the katana is uniquely known for, the swordsmith removes the blade from the coals and immediately quenches the blade in a water trough. The different concentrations of carbon in the two

sections of the blade cause the blade's curve. This process is so difficult that one out of three swords is ruined during this process. The blade then goes through a series of polishing stones and is decorated with different metals and is fitted with a handle for completion.

Procedure

This project consisted of three different parts. The characterization of two local iron oxide concentrates and one commercial blast furnace pellet was the first part. The next part consisted of creating pellets to be reduced to iron. The last part involved running a series of experiments to reduce the iron oxide to metallic iron.

Characterization of Ores

Two different ores and one commercial pellet were characterized to determine what material would be best for producing iron or steel in a primitive blast furnace. The sample from Pacer Minerals (Custer, SD) was analyzed for particle size distribution and mica content. There were four different stages of the Pacer sample. The bulk material was the starting point of the ore. Then, the ore was crushed in a jaw crusher to reduce the particle size and to separate the mica from the magnetite. The ore was then separated using a shaker table where smaller finer particles gravitate towards the end of the table and are deposited into pans according to size and shape. After the shaker table the magnetite was separated from the mica using a magnetic separator. The aforementioned agglomeration group did this work.

The four different stages were each analyzed for particle size distribution. Stages were labeled by name by what the last process of separation they had gone through. The

stages were named: after magnetic separator, after shaker table, after crusher, and drum material. To gather a representative sample of the bulk material, the ore was put through a series of Jones splitters. After magnetic separator, after shaker table, and after crusher were all split three times with the largest Jones splitter. They were then split three times with the medium Jones splitter. Finally, they were split two times with the small Jones splitter. The drum material was split three times with the largest Jones splitter and then split four times with the medium Jones splitter. Due to particle size the drum material could not be split with the smallest Jones splitter. A sieve analysis was then performed on the representative samples of each stage of the material to determine particle size for each step of the process. Approximately 1000 grams of ore was poured in the top of a series of sieves. The sieves consisted of 10, 16, 20, 50, 80, 100, 140, and 200 U.S. Standard mesh. The shaker was run for 10 minutes in each of three trials for each mineral sample. The ore in each of the sieves was weighed and recorded in a table.

A representative sample was also taken of each stage of the material to determine the mica content of the material. Quantitative x-ray diffraction analysis was performed on each sample.

To better understand what material would be best for reducing the iron ore or pellet to iron, an experiment was conducted to try and reduce each material to iron in a muffle furnace. Three graphite crucibles were purchased and were each filled with one of the different materials and carbon. Crucible 1 contained the Pacer material and carbon layered in about 12-14 alternating layers. Crucible 2 contained the commercially available pellets from Cleveland Cliffs and carbon. The carbon and pellets were layered alternately although the difference in particle size of the pellets compared to the carbon

caused a homogenous mixture of pellets and carbon throughout the crucible. Crucible 3 contained ore that remained after precipitation from the Homestake dewatering process and carbon. This material was mixed throughout the crucible. The amount of carbon added to each crucible was dependent on stoichiometric calculations to determine the amount of carbon needed to fully reduce the ore in the crucible with ten-percent excess. Not all of the ore was able to fit in the third crucible, so there was a greater excess of carbon than was actually needed. The furnace was heated to $1000^{o}C$ and the crucibles were placed inside of the furnace. They were kept inside the furnace for four hours. They were then taken out of the furnace and placed inside of a steel container where they would cool in a nitrogen atmosphere. This was accomplished by placing a crucible full of liquid nitrogen in the steel container as well. After the crucibles had cooled to room temperature, the ore was taken out, weighed, and analyzed. Some samples were put up to a grinding wheel to see if they sparked which would indicate metallic iron present in the material. Magnets were also placed near the material to test for the presence of iron. Some of the pellets were ground halfway, mounted in Bakelite, and polished. Samples were also sent for a quantitative x-ray diffraction analysis.

Agglomeration

A number of different methods were tried before one worked well. First, a ten-gallon Nalgene container with the bottom cut off was used as a tumbler to ball the fine ore into small pellets. At first, water was poured on top of the ore in the container with a graduated cylinder, but that led to the ore balling up in large clumps. A squirt bottle was tried next. That gave better results, but still had some of the same problems. A spray bottle produced the best results because the ore was thoroughly wetted and agglomerated

into pellets ranging from 3-10 mm. These pellets once dried did not hold up under some

drop tests from shoulder height. Experiments were then conducted by mixing different

binders into the ore. Bentonite was used as a binder but the pellets were not strong

enough in the drop tests. Flour and sugar were also used as binders, but when the pellets

were dried and sintered they fell apart. Polyvinyl Alcohol (PVA) was also used as a

binder, but the PVA would not dissolve in water making it ineffective as a binder for the

ore.

Work was then done looking into other methods of pelletization. One of the

methods looked into involved using a meat grinder and putting the iron ore sludge

through it and small cylindrical pellets would form. Initial experimentation was done

with the meat grinder to see how the pellets would adhere and to see whether or not the

ore would easily feed through the meat grinder. Initially, eight mass percent of bentonite

was added to the iron ore sludge and mixed thoroughly. The mixture was then put

through the meat grinder, and the pellets were dried. From those results, it was

determined that the moisture content of the wet iron ore sludge and burden was based on

the mass of the ore and not the mass of the water and ore. The mass of the wet sludge was

taken and then the sludge was put into an induction furnace set at $300^{o}C$. After the sludge

had thoroughly dried, the mass of the dry ore was taken. This was done three different

times. The percent of water in the sludge was figured out by the mass lost while drying.

Table 1 lists the analysis of the ore and water in the sludge for each of the three trials and

the averages for each.

Table 1: Analysis of Moisture Content in Iron Ore Sludge

	Initial Weight of Sludge (g)	Weight of dried ore (g)	Percent Water	Percent Ore
Trial 1	402.2	104.4	74.04	25.96
Trial 2	258.4	64.2	75.15	24.85
Trial 3	270.9	62.2	77.04	22.96
Average			75.41	24.59

Knowing the moisture content in the iron ore sludge, pellets containing varying amounts of bentonite and carbon were created. Some pellets were sintered and others were not, depending on the strength of the pellets. Table 2 shows the different pellets created and the sintering process each went through.

Table 2: Pellet Composition and Sintering Process for All Trials

Pellet Composition						Sintering Process		
Trial Number	Pellet Description	Mass Wet Sludge	Mass Ore (g)	Bentonite Added (g)	Carbon Added (g)	Furnace or Forge	Temperature (^{o}C)	Time (minutes)
1	No Carbon, No Bentonite	88.7	21.6	0	0	Forge	900-1100	30
2	No Carbon, 3% Bentonite	397.7	97.8	2.9	0	Forge	900-1100	30
3	Carbon, 3% Bentonite	427.6	105.2	3.2	26.1	Furnace	700	75
4	No Carbon, 3% Bentonite	500.9	123.2	3.7	0	Furnace	750	75
5	No Carbon, 3% Bentonite	1099.3	270.3	8.1	0	Furnace	750	90

Direct Reduction

A series of experiments listed in Table 10 were completed using a quartz tube furnace, shown in Figure 2, in an attempt to reduce the iron ore to iron. The furnace

consists of refractory brick inside of a steel casing where the right circle is in Figure 2. A glass tube runs through the center of the furnace where an alumina boat filled with pellets is pushed into the center where it can be heated. A cooling system is placed on either end of the glass tube to prevent the tube from melting the corks that stop the gas inside of the furnace from escaping. This cooling system can be seen in Figure 2 and is represented by the three triangles. Argon or carbon monoxide gas is attached to one end of the tube to create a reducing atmosphere inside of the tube. On the other end of the tube, the gas runs through a rubber hose into a bubbler that allows the user to see how fast the gas is flowing through the tube. The bubbler can be seen in Figure 2 and is shown by the square. In all of the experiments the furnace was started, the temperature rose to $200^{o}C$, and then the temperature then rose evenly over 30 minutes to $1000^{o}C$. The furnace then needs about 1 hour to calibrate to the desired temperature. Once that hour was up, the samples were inserted into the furnace and either carbon monoxide or argon gas was turned on depending on the type of pellets in the furnace. Once the sample had been in the furnace for the required amount of time, the furnace was shut off and the gas was kept on to keep a reducing atmosphere in the furnace. In the first two trials, the carbon monoxide gas was allowed to flow and the boat was left in the middle of the furnace. In later experiments, once the furnace was shut off, the gas was switched to argon in order to save the carbon monoxide for future experiments and to keep an inert atmosphere in the tube. The boat was also pulled to one end of the tube in order to let it cool faster than when it was in the middle of the furnace.

Figure 2: Quartz Tube Furnace Setup for Reaction Kinetics Experiments

A test was also conducted in a muffle furnace to try and reduce and then melt the pellets. The muffle furnace was turned on and was heated up to around 1000°C to 1075°C. Three crucibles were then placed in the furnace for six hours. The contents of the crucibles are listed in Table 3. Once the crucibles had been in the furnace for six hours the temperature was increased to around 1200°C and held for one hour to try and melt the pellets. The pellets were not melted after we took them out after six hours, so carbon was poured over the top of the pellets to keep them insulated from the oxygen atmosphere and put back in the furnace to cool overnight.

Table 3: Contents of Crucibles in Muffle Furnace Test

Crucible #	Type Pellet	Mass Pellets	Mass Carbon
1	Non Carbon, 3% Bentonite	110.8	27.5
2	Non Carbon, 3% Bentonite	107.8	26.8
3	Carbon, 3% Bentonite	108.6	Carbon Present in Pellets

Results

This section details the results of each of the three parts to this project: characterization of the ores, agglomeration, and direct reduction.

Characterization of Ores

Table 4 through Table 7 detail the particle size distribution for each of the four different stages in the process of extracting the mica from the magnetite in the Pacer sample. Graphs of the particle size distribution for Table 4 through Table 7 can be seen in Appendix A.

Table 4: Average Particle Size Distribution for After Magnetic Separation

	After Magnetic Separation					
	U.S. Standard Mesh	Diameter (μm)	Mass (g)	Mass % Retained	Cumulative % Retained	Cumulative % Passing
+	10	2000	0.00	0.00	0.00	100.00
+	16	1180	0.67	0.07	0.07	99.93
+	20	850	1.60	0.16	0.23	99.77
+	50	300	36.20	3.62	3.85	96.15
+	80	180	15.60	1.56	5.41	94.59
+	100	150	63.07	6.31	11.72	88.28
+	140	106	360.90	36.11	47.83	52.17
+	200	75	271.00	27.12	74.95	25.05
-	200	0	250.33	25.05	100.00	0.00
		Total	999.37	100.00		

Table 5: Average Particle Size Distribution for After Shaker Table

	After Shaker Table Average					
	U.S. Standard Mesh	Diameter (μm)	Mass (g)	Mass % Retained	Cumulative % Retained	Cumulative % Passing
+	10	2000	0.37	0.04	0.04	99.96
+	16	1180	0.67	0.07	0.10	99.90
+	20	850	9.00	0.90	1.00	99.00
+	50	300	635.17	63.55	64.56	35.44
+	80	180	202.07	20.22	84.78	15.22
+	100	150	55.20	5.52	90.30	9.70
+	140	106	49.40	4.94	95.24	4.76
+	200	75	17.80	1.78	97.02	2.98
-	200	0	29.73	2.98	100.00	0.00
		Total	999.40	100.00		

Table 6: Average Particle Size Distribution for After Crusher

	After Crusher Average					
	U.S. Standard Mesh	Diameter (µm)	Mass (g)	Mass % Retained	Cumulative % Retained	Cumulative % Passing
+	10	2000	2.57	0.26	0.26	99.74
+	16	1180	35.97	3.60	3.86	96.14
+	20	850	98.17	9.82	13.68	86.32
+	50	300	607.43	60.79	74.47	25.53
+	80	180	133.30	13.34	87.80	12.20
+	100	150	40.73	4.08	91.88	8.12
+	140	106	36.93	3.70	95.58	4.42
+	200	75	16.43	1.64	97.22	2.78
-	200	0	27.77	2.78	100.00	0.00
		Total	999.30	100.00		

Table 7: Average Particle Size Distribution For Drum Material

	Drum Material Average					
	U.S. Standard Mesh	Diameter (µm)	Mass (g)	Mass % Retained	Cumulative % Retained	Cumulative % Passing
+	10	2000	81.67	8.17	8.17	91.83
+	16	1180	311.20	31.12	39.29	60.71
+	20	850	230.17	23.02	62.30	37.70
+	50	300	243.47	24.35	86.65	13.35
+	80	180	78.30	7.83	94.48	5.52
+	100	150	21.53	2.15	96.63	3.37
+	140	106	15.10	1.51	98.14	1.86
+	200	75	6.40	0.64	98.78	1.22
-	200	0	12.17	1.22	100.00	0.00
		Total	1000.00	100.00		

Table 8 shows the weight percent of the different minerals present in the Pacer ore at each step of processing the ore. This information was gathered by doing a quantitative analysis from profile-fitted peaks. The calculations are based on peak area using preferred orientation correction and the Brindley correction at five microns. The XRD graphs and quantitative analysis sheets can be seen in Appendix B.

Table 8: Quantitative Analysis of Pacer Material

Sample	Weight Percent Magnetite	Weight Percent Muscovite	Weight Percent Quartz	Weight Percent Hematite
After Magnetic Separator	50.6	38.2	7.7	3.5
After Shaker Table	31	63.5	5.5	0
After Crusher	17.2	65.9	16.9	0
Drum Material	30.4	62.1	7	0.5

Initial XRD of the Homestake ore showed that the ore was amorphous or the particles were too small to create an XRD pattern. Under the scanning electron microscope very small crystals could be seen. Because of the small crystals present the iron ore was determined to be limonite ($FeO(OH) \cdot nH_2O$). The Homestake iron ore was determined to be about 96% hematite by analyzing the iron ore with a florescence scan in the SEM.

The results from the three samples in the muffle furnace are as follows. The Pacer sample was put into the furnace as a powder and when it was removed it came out of the crucible as one block as seen in Figure 2. When the bottom of the block was held to the grinding wheel, it sparked indicating the presence of metallic iron. When a magnet was touched to the Cleveland Cliffs pellets and the Homestake material, the material was

attracted to it. This indicated that the ore had at least reduced to magnetite if not metallic iron. The Cleveland Cliffs pellets and the Homestake material can be seen in Figures 3 and 4 respectively. A quantitative analysis was done on the Cleveland Cliffs pellets. The pellets were composed of 6.4 wt% metallic iron, 77.4 wt% wuestite (FeO), and 16.2 wt% kirschsteinite ($CaFe^{+2}SiO_4$). The XRD graphs and analysis can be seen in Appendix C. A quantitative analysis was not done on the Homestake or pacer, although it is assumed that all the Homestake ore was reduced to pure iron because only one peak showed up on the XRD scan.
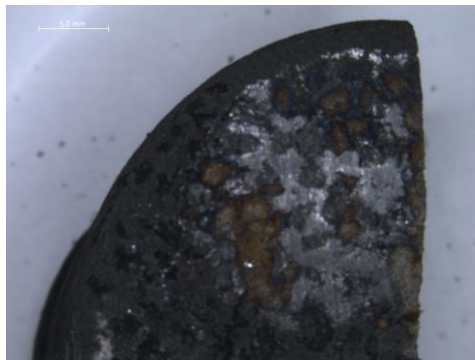

Figure 3: Pacer Sample After First Muffle Furnace Test

Figure 4: Cleveland Cliffs Pellets after First Muffle Furnace Test

Figure 5: Homestake Material after First Muffle Furnace Test

Agglomeration

Table 9 lists the different types of pellets created and has a description of the strength of each.

Table 9: Strength of Pellets Created

Trial Number	Pellet Description	Description of Strength of the Pellet
1	No Carbon, No Bentonite Pellets	When dropped from head height onto concrete, pellets broke into smaller pieces. Did not crumble. Could squeeze between your fingers and they would not break apart easily.
2	No Carbon, 3% Bentonite Pellets	After sintering looked as if they had reduced somewhat. These pellets were very strong. They could be dropped from head height and would not break apart.
3	Carbon, 3% Bentonite Pellets	These carbon pellets were extremely weak. When pressed on a bit, they crumbled apart into small pieces. These pellets before sintering had more strength than after sintering.
4	No Carbon, 3% Bentonite Pellets	When dropped from head height onto concrete, pellets broke into smaller pieces. Did not crumble. Could squeeze between your fingers and they would not break apart easily.
5	No Carbon, 3% Bentonite Pellets	When dropped from head height onto concrete, pellets broke into smaller pieces. Did not crumble. Could squeeze between your fingers and they would not break apart easily.

Direct Reduction

Table 10 describes the type of pellets used in the quartz tube furnace along with the gas used in each trial and the time the pellets were in the furnace. It also shows the % mass lost while in the furnace.

Table 10: Description of Pellets and Experiments Run, and Mass Reduction in Quartz Tube Furnace

Trial #	Description of Pellets	Time in Furnace	Gas Used	% Mass Lost
1	Non Carbon, About 32% bentonite pellets	3hrs 20min	Carbon Monoxide	12.15%
2	Non Carbon, About 32% bentonite pellets	2hrs	Carbon Monoxide	10.38%
3	Non Carbon, No Bentonite Pellets	4hrs	Carbon Monoxide	14.85%
4	Carbon, 3% Bentonite Pellets	6hrs	Argon	26.74%
5	Non Carbon, 3% Bentonite Pellets	6hrs	Carbon Monoxide	20.56%

Discussion

Performing the initial characterization tests on the different ores provided a basis for determining which material should be used to create iron. The XRD analysis of the four steps of the Pacer material showed that there was still 45.9% material that was not iron ore. The crushing of the ore separated some of the mica from the magnetite, but the shaker table did not seem to do much in reducing the amount of mica in the ore. This other material is essentially all impurities and affects how the Pacer material would reduce. There is also an inconsistency with the data for the after crusher sample. The data shows only 17.2% magnetite in the ore, where the drum had 30.4%. An unrepresentative sample must have been taken and placed in the XRD machine. The first muffle furnace test that was performed showed what material should be focused on in the pelletization process. A quantitative XRD analysis was not done on the Homestake ore because only iron showed up as a peak. Some noise caused iron oxide to show up but not a significant amount to note. A quantitative analysis was not done for the Pacer material either. This was due to a large unknown peak being present. It was assumed to be graphite. Graphite

crucibles were used and some of the graphite could have fallen off the side and gotten into the sample. After examining the wustite and iron peaks on the XRD graph, one can see that the wustite peak is much greater than the iron one. Iron is more than two times as good of a reflector than wustite is, which means that even less iron is present than wustite is.

When pellets were first made, the ore was dried and ground to a fine powder. When water was added to the ore, the consistency of the ore was never the same than that of the initial material. The consistency of the initial material was a sticky sludge where the consistency of the re-hydrated ore was more of a wet grainy texture. The pellets formed from the ground, dried ore would not hold together nearly as the initial ore dried in pellet form. When first making pellets with the meat grinder, 8% bentonite was added by mass not taking into account the water content in the iron ore sludge. Once this was realized, calculations were performed and around 32% by mass was actually added to the dry ore. This posed problems with the reduction of the ore in the quartz tube furnace. It is believed that the bentonite hindered the reduction of the ore and did not allow the ore to reduce as much as it could of.

When iron oxide is directly reduced by carbon or carbon monoxide, the oxygen that is bonded to the iron comes off of the iron and is converted to carbon dioxide as seen in Equations 1 through 4. In the experiments ran in the tube furnace the carbon dioxide should have left the tube furnace and resulted in a loss of mass. This loss of mass can be correlated to the amount of reduction the ore went through. The lower the percent mass loss the less reduced the material should be and the higher the percent mass loss the more reduced the material should be.

$$Fe_2O_3 + CO \rightarrow 2Fe_3O_4 + CO_2 \qquad \text{(Equation 1)}$$

$$Fe_3O_4 + CO \rightarrow 2FeO + CO_2 \qquad \text{(Equation 2)}$$

$$FeO + CO \rightarrow Fe + CO_2 \qquad \text{(Equation 3)}$$

$$Fe_2O_3 + 3C \rightarrow 2Fe + 3CO \qquad \text{(Equation 4)}$$

In the final muffle furnace test, it is believed that the pellets were not heated sufficiently to melt them or that not enough carbon was added in order to lower the melting point of the iron. Once the carbon for reducing the ore is used, the excess carbon can diffuse into the iron to produce steel. By having a eutectic amount of carbon present in the steel the melting point can be significantly reduced from 1539°C to 1147°C as seen in Figure 6. Having a greater than 10% excess of carbon of the crucibles could have further lowered the melting point of the iron.
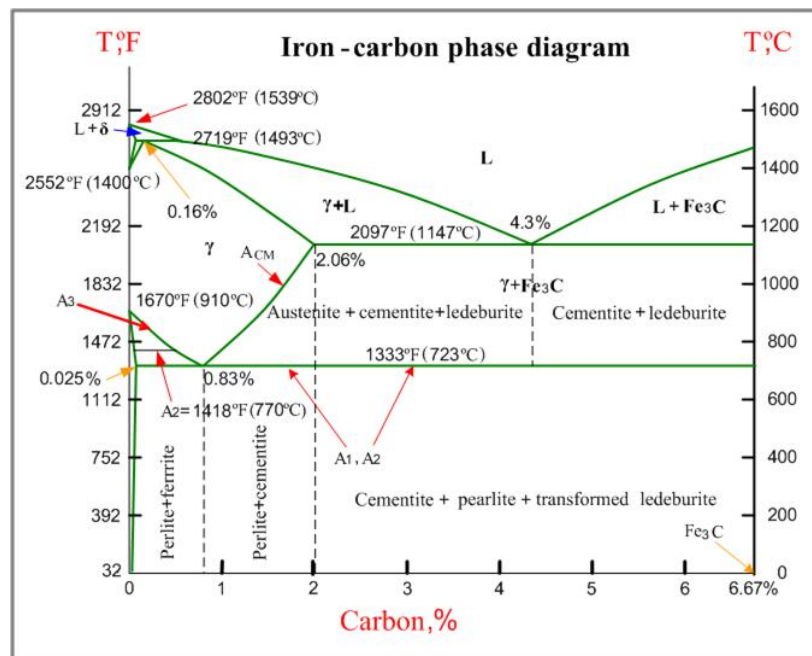

Figure 6: Iron Carbon Phase Diagram

Conclusions

Strong pellets containing small amounts of bentonite could be formed using a meat grinder. These pellets did not reduce well, but further experimentation could be conducted on making strong quick reducing pellets. Experimentation could be done on reducing the bentonite content of the pellets. The results of the direct reduction were inconclusive. Further experimentation should be done in this area.

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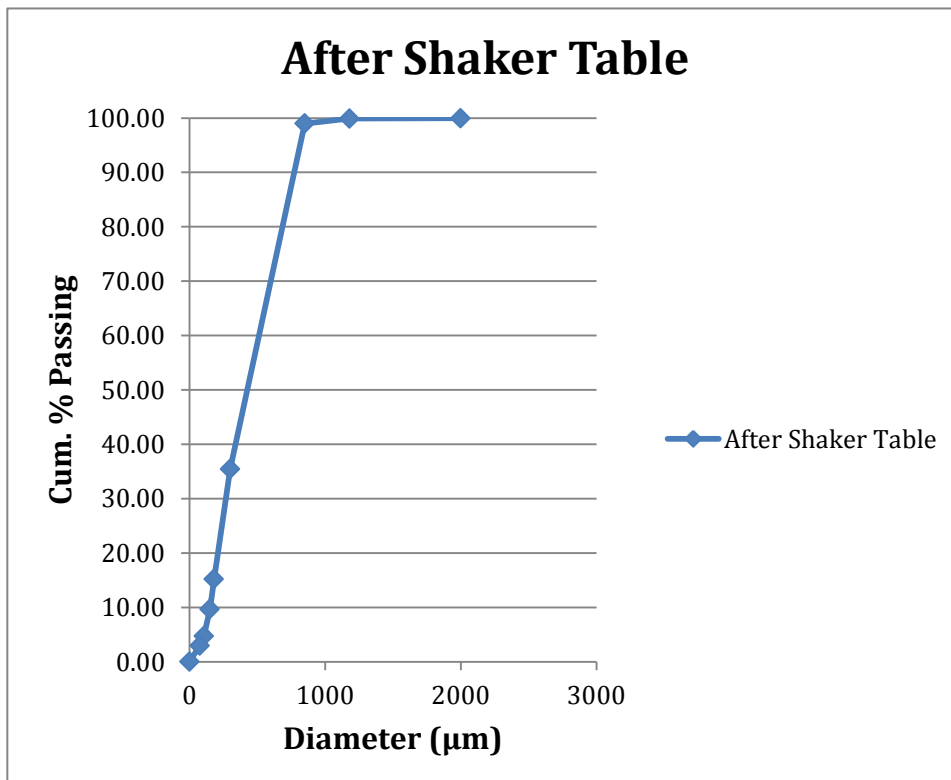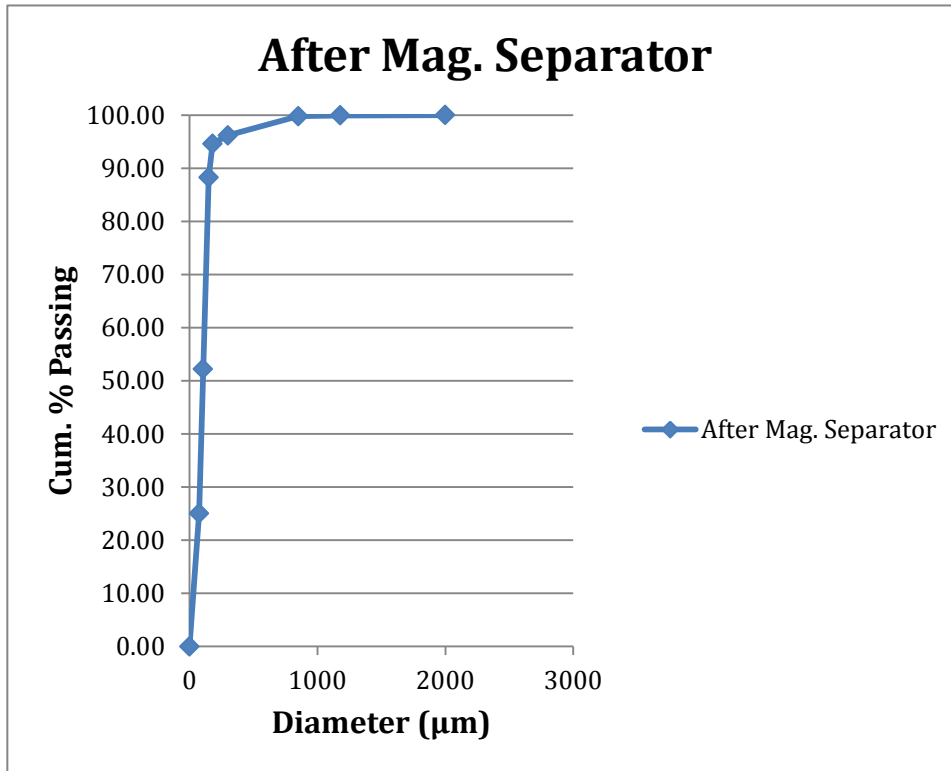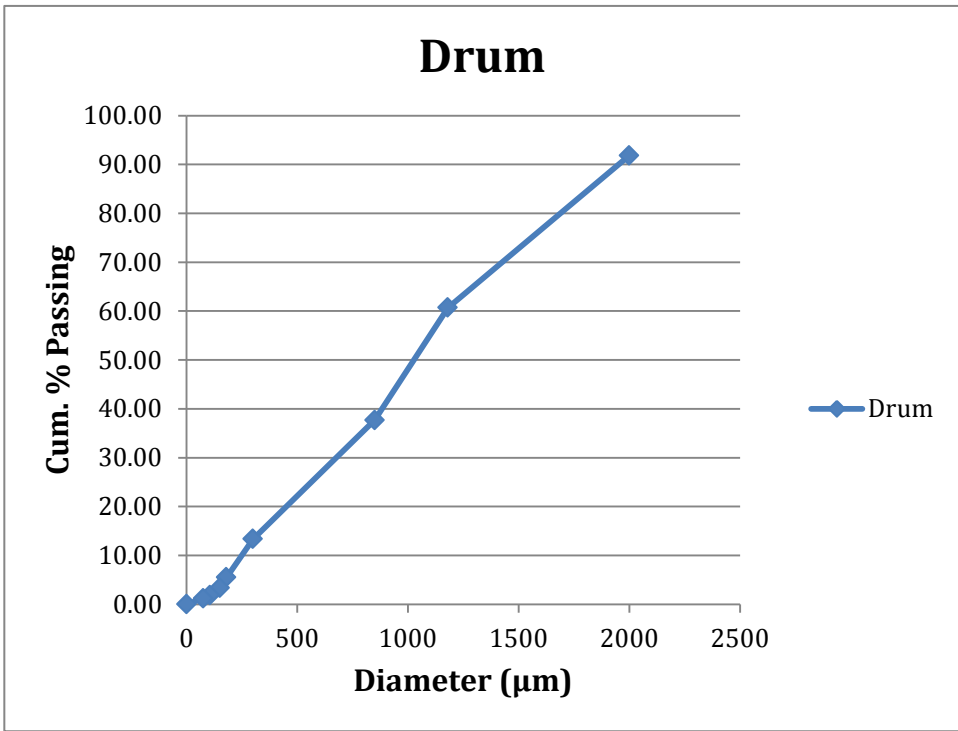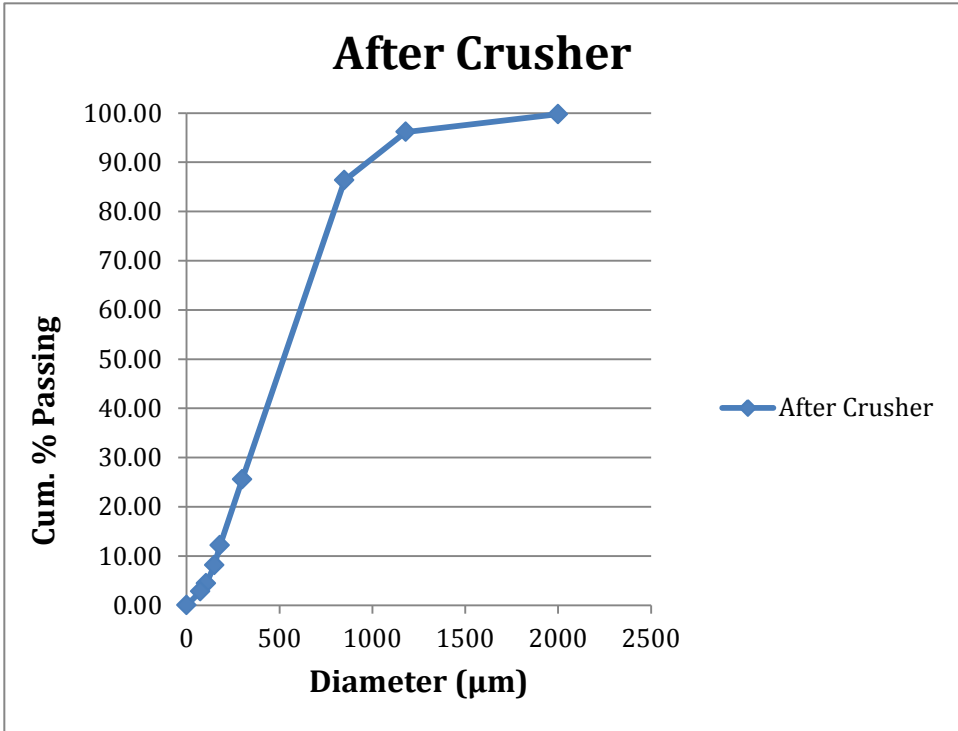## Acknowledgements

Appendices

Appendix A: Graphs of Particle Size Distribution for Pacer Material

After Mag. Separator

After Shaker Table

After Crusher

Cum. % Passing vs Diameter (μm)

Drum

Cum. % Passing vs Diameter (μm)

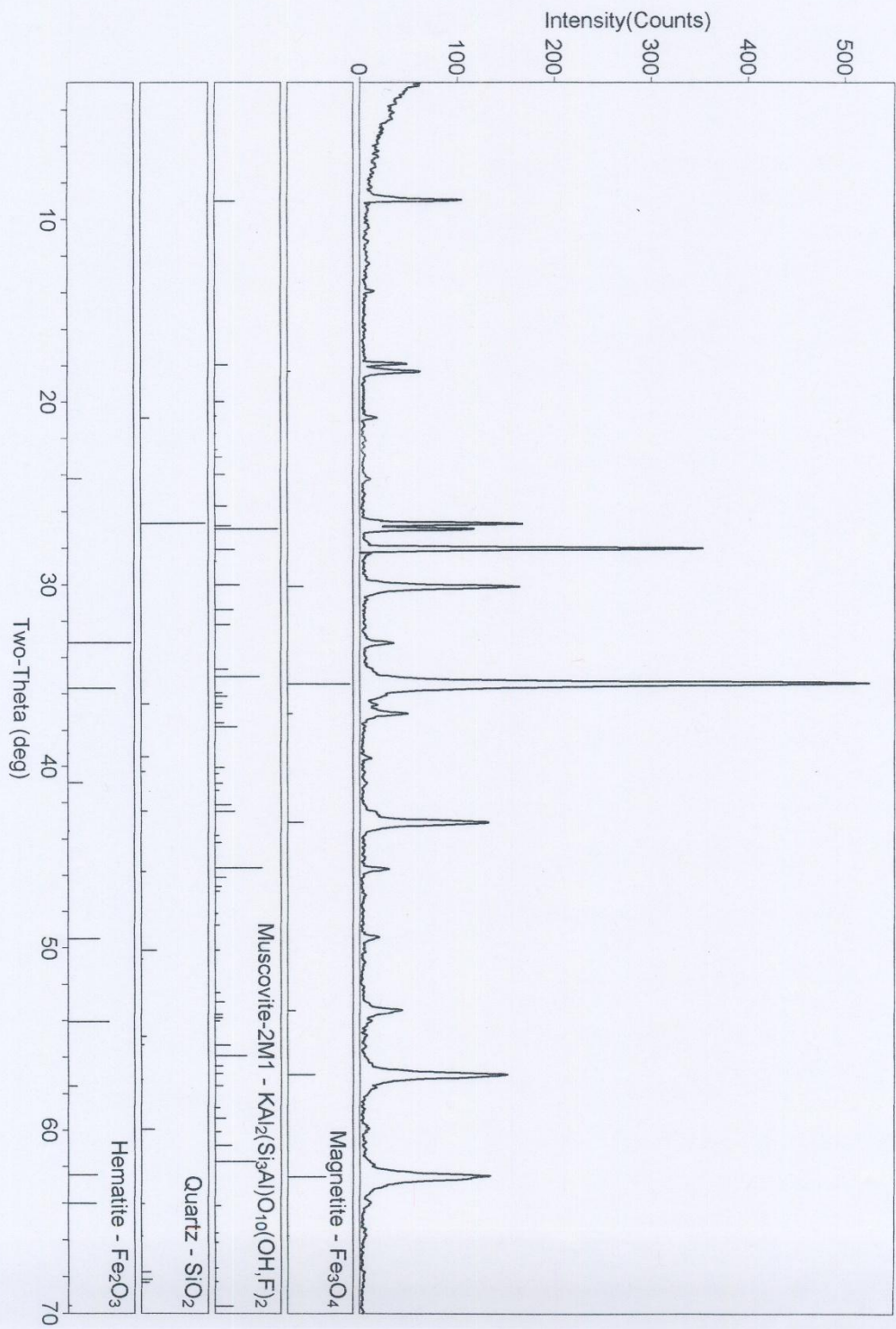**Appendix B:** Quantitative analysis and graphs for XRD for Pacer Ore

Quantitative Analysis from Profile-Fitted Peaks

FILE: [089-0448.raw] After Mag Sep
SCAN: 2.5/70.0/0.02/0.6(sec), Cu(40KV,40mA), I(max)=525, 06/09/09 01:41p
PROC: [New Quantitative Analysis]

Phase ID (4)	RIR	Wt%	Wt(n)%	Vol(n)%	#L	I%-I(r)	POC
Magnetite - Fe_3O_4	5.13	50.6 (16.0)	50.6 (16.0)	36.4 (13.6)	6	6.7	<None>
Muscovite-2M1 - $KAl_2(Si_3Al)O_{10}(OH,F)_2$	0.52	38.2 (12.1)	38.2 (12.1)	50.3 (18.8)	3	6.8	<None>
Quartz - SiO_2	3.41	7.7 (2.4)	7.7 (2.4)	10.8 (4.0)	1	0.1	<None>
Hematite - Fe_2O_3	3.24	3.5 (1.1)	3.5 (1.1)	2.5 (0.9)	1	0.0	<None>

NOTE: Calculation Using: Peak Area, POC: Preferred Orientation Correction, Brindley Correction = 5.0 microns

2-Theta	FWHM	Height	H%	Area(a1)	A%	I(r)	I(p)	I%-I(r)	(h k l)
18.314 (0.018)	0.194 (0.025)	42 (4)	11.1	582 (64)	8.6	5.1	5.1	3.5	(1 1 1)
30.091 (0.005)	0.242 (0.006)	112 (2)	29.4	1752 (43)	25.8	24.8	24.8	1.0	(2 2 0)
35.431 (0.008)	0.257 (0.016)	381 (15)	100.0	6802 (359)	100.0	100.0	100.0	0.0	(3 1 1)
43.094 (0.010)	0.242 (0.026)	102 (7)	26.8	1865 (157)	27.4	24.8	24.8	2.6	(4 0 0)
56.963 (0.013)	0.257 (0.046)	124 (13)	32.5	2453 (327)	36.1	43.1	43.1	-7.0	(5 1 1)
62.528 (0.007)	0.294 (0.025)	108 (5)	28.4	2440 (154)	35.9	62.0	62.0	-26.1	(4 4 0)
8.974 (0.012)	0.183 (0.018)	62 (5)	69.6	688 (72)	100.0	100.0	100.0	0.0	(0 0 2)
17.881 (0.019)	0.123 (0.016)	32 (5)	36.2	213 (39)	30.9	20.0	20.0	10.9	(0 0 4)
26.934 (0.028)	0.131 (0.043)	89 (20)	100.0	622 (231)	90.3	100.0	100.0	-9.7	(0 0 6)
26.674 (0.019)	0.136 (0.025)	129 (22)	100.0	1054 (219)	100.0	100.0	100.0	0.0	(1 0 1)
33.180 (0.016)	0.175 (0.044)	24 (3)	100.0	310 (60)	100.0	100.0	100.0	0.0	(1 0 4)

Intensity(Counts)

Two-Theta (deg)

Magnetite - Fe₃O₄

Muscovite-2M1 - KAl₂(Si₃Al)O₁₀(OH,F)₂

Quartz - SiO₂

Hematite - Fe₂O₃

Quantitative Analysis from Profile-Fitted Peaks

FILE: **[089-0447.raw] After Shaker Table**

SCAN: 2.5/70.0/0.02/0.6(sec), Cu(40kV,40mA), I(max)=1101, 06/09/09 04:01p

PROC: [New Quantitative Analysis]

Phase ID (3)

☐ Magnetite - Fe_3O_4

☐ Muscovite-2M1 - $KAl_2(Si_3Al)O_{10}(OH,F)_2$

☐ Quartz - SiO_2

	RIR	Wt%	Wt(n)%	Vol(n)%	#L	I%-I(r)	POC
	5.13	31.0 (3.3)	31.0 (3.3)	19.6 (2.6)	4	2.6	<None>
	0.52	63.5 (6.7)	63.5 (6.7)	73.5 (9.7)	3	12.9	<None>
	3.41	5.5 (0.6)	5.5 (0.6)	6.8 (0.9)	1	0.0	<None>

NOTE: Calculation Using: Peak Area, POC: Preferred Orientation Correction, Brindley Correction = 5.0 microns

2-Theta	FWHM	Height	H%	Area(a1)	A%	I(r)	I(p)	I%-I(r)	(h k l)
18.286 (0.012)	0.158 (0.014)	86 (6)	9.7	846 (76)	8.7	5.1	5.1	3.6	(1 1 1)
30.052 (0.005)	0.179 (0.007)	184 (6)	20.7	2127 (89)	21.9	24.8	24.8	-2.9	(2 2 0)
35.417 (0.001)	0.161 (0.003)	888 (12)	100.0	9700 (159)	100.0	100.0	100.0	0.0	(3 1 1)
43.050 (0.015)	0.166 (0.034)	253 (39)	28.5	2766 (536)	28.5	24.8	24.8	3.7	(4 0 0)
8.842 (0.004)	0.253 (0.005)	279 (4)	90.3	4083 (86)	100.0	100.0	100.0	0.0	(0 0 2)
17.831 (0.012)	0.151 (0.013)	79 (7)	25.5	750 (71)	18.4	20.0	20.0	-1.6	(0 0 4)
26.900 (0.007)	0.149 (0.007)	308 (15)	100.0	2568 (154)	62.9	100.0	100.0	-37.1	(0 0 6)
26.555 (0.015)	0.203 (0.016)	156 (10)	100.0	2000 (159)	100.0	100.0	100.0	0.0	(1 0 1)

Intensity(Counts)

Two-Theta (deg)

Magnetite - Fe₃O₄

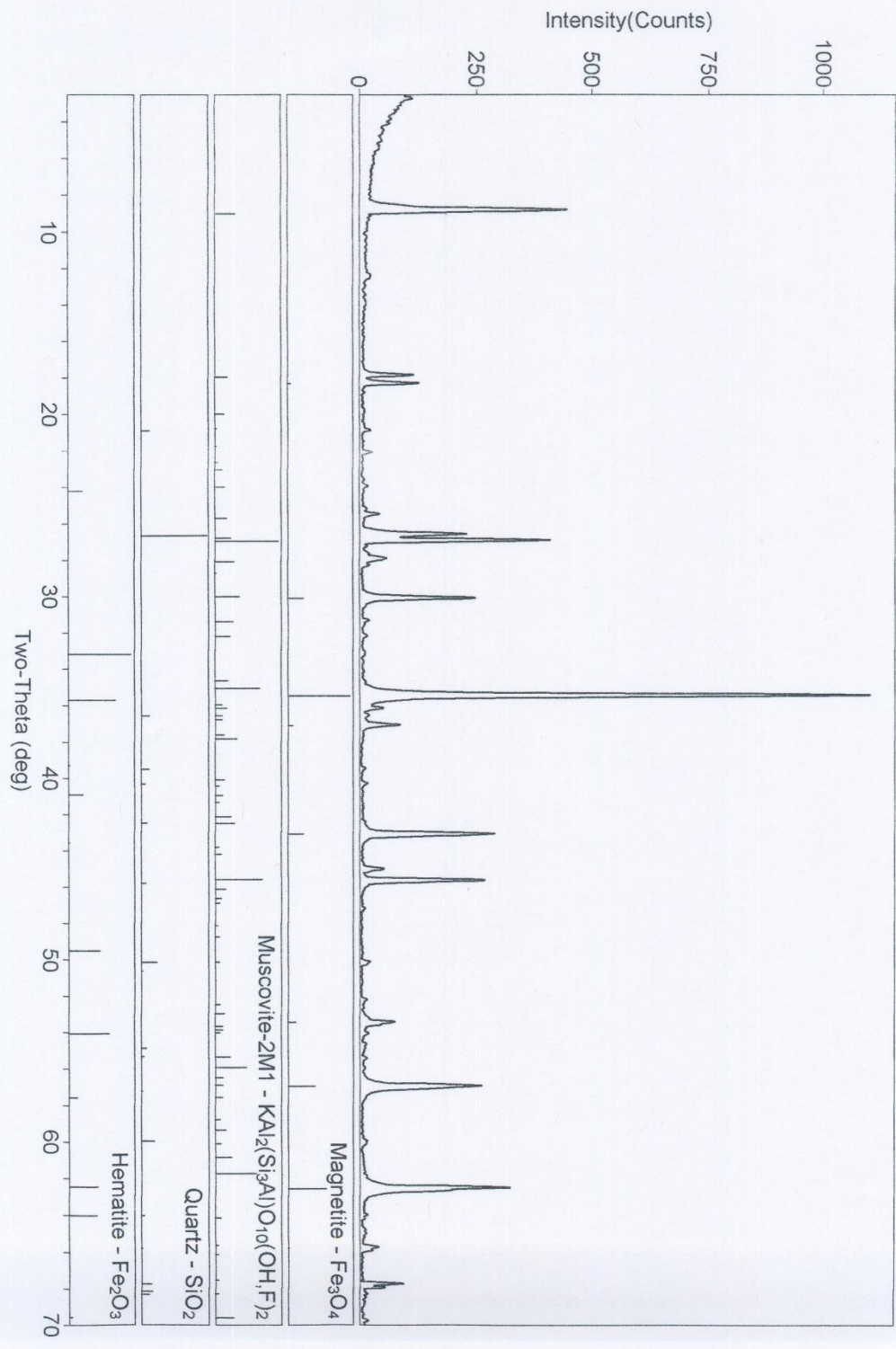Muscovite-2M1 - KAl₂(Si₃Al)O₁₀(OH,F)₂

Quartz - SiO₂

Hematite - Fe₂O₃

Quantitative Analysis from Profile-Fitted Peaks

FILE: [089-0446.raw] After Crusher
SCAN: 2.5/70.0/0.02/0.6(sec), Cu(40kV,40mA), I(max)=1611, 06/09/09 03:21p
PROC: [New Quantitative Analysis]

Phase ID (3)

		RIR	Wt%	Wt(n)%	Vol(n)%	#L	I%-I(r)	POC
☑	Magnetite - Fe_3O_4	5.13	17.2 (1.1)	17.2 (1.1)	10.0 (0.8)	5	3.6	<None>
☑	Muscovite-2M1 - $KAl_2(Si_3Al)O_{10}(OH,F)_2$	0.52	65.9 (4.2)	65.9 (4.2)	70.6 (5.7)	4	13.4	<None>
☐	Quartz - SiO_2	3.41	16.9 (1.1)	16.9 (1.1)	19.4 (1.6)	1	0.0	<None>

NOTE: Calculation Using: Peak Area, POC: Preferred Orientation Correction, Brindley Correction = 5.0 microns

2-Theta	FWHM	Height	H%	Area(a1)	A%	I(r)	I(p)	I%-I(r)	(h k l)
18.306 (0.013)	0.152 (0.012)	128 (10)	24.6	1093 (111)	14.3	5.1	5.1	9.2	(1 1 1)
30.076 (0.008)	0.166 (0.015)	166 (11)	31.9	1893 (154)	24.8	24.8	24.8	0.0	(2 2 0)
35.420 (0.003)	0.219 (0.005)	522 (8)	100.0	7629 (153)	100.0	100.0	100.0	0.0	(3 1 1)
37.043 (0.006)	0.187 (0.011)	97 (4)	18.6	1198 (66)	15.7	8.0	8.0	7.7	(2 2 2)
43.046 (0.003)	0.154 (0.010)	173 (7)	33.1	1995 (103)	26.2	24.8	24.8	1.4	(4 0 0)
8.864 (0.004)	0.217 (0.004)	650 (11)	100.0	7813 (176)	100.0	100.0	100.0	0.0	(0 0 2)
17.844 (0.010)	0.138 (0.010)	197 (15)	30.3	1712 (143)	21.9	20.0	20.0	1.9	(0 0 4)
20.805 (0.280)	0.199 (?)	14 (?)	2.1	201 (?)	2.6	3.0	3.0	-0.4	(1 1 1)
26.898 (0.006)	0.133 (0.007)	489 (24)	75.2	3806 (230)	48.7	100.0	100.0	-51.3	(0 0 6)
26.637 (0.003)	0.164 (0.005)	1180 (23)	100.0	11437 (364)	100.0	100.0	100.0	0.0	(1 0 1)

Intensity(Counts)

Two-Theta (deg)

Magnetite - Fe₃O₄

Muscovite-2M1 - KAl₂(Si₃Al)O₁₀(OH,F)₂

Quartz - SiO₂

Hematite - Fe₂O₃

Quantitative Analysis from Profile-Fitted Peaks

FILE: **[089-0446.raw] After Crusher**
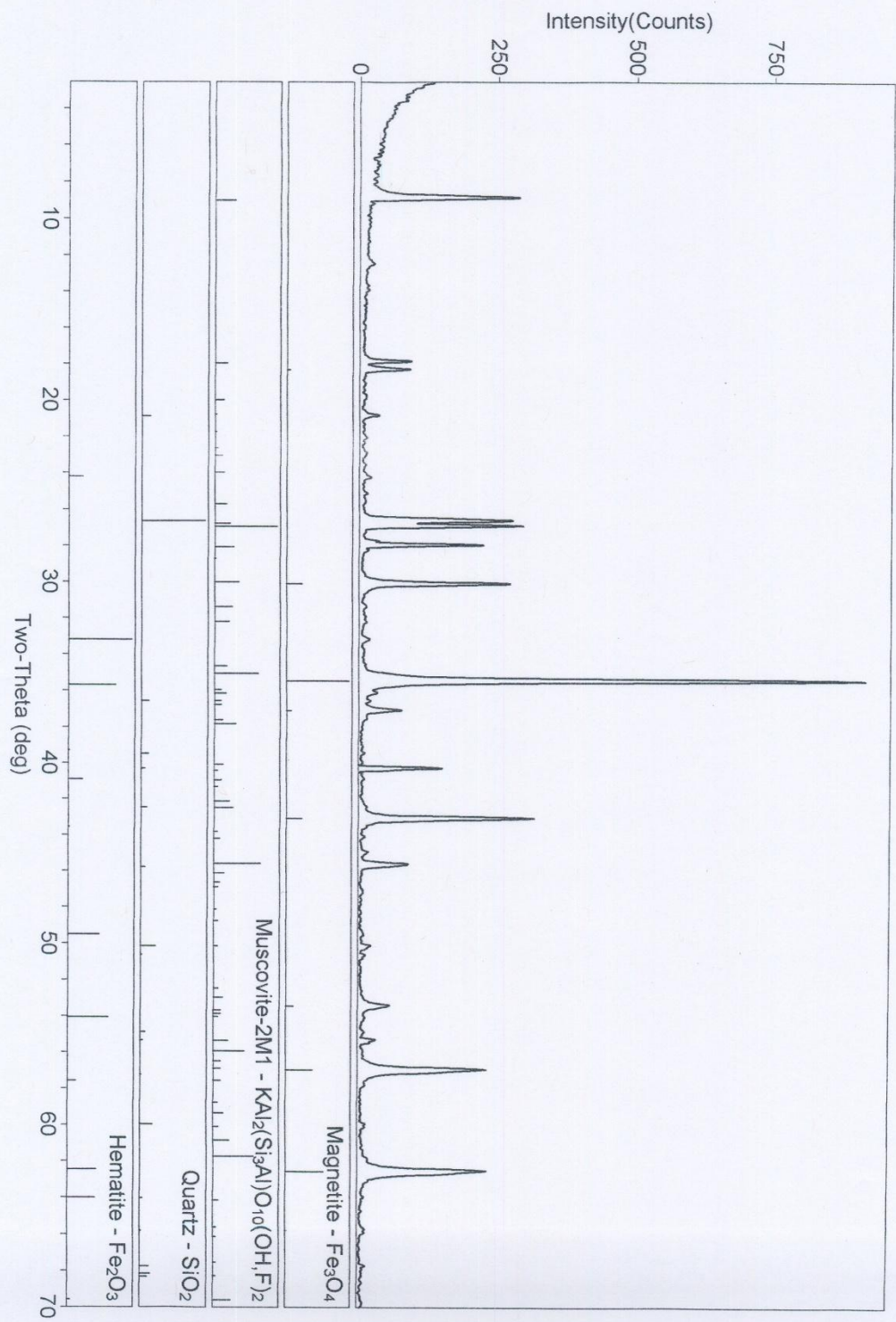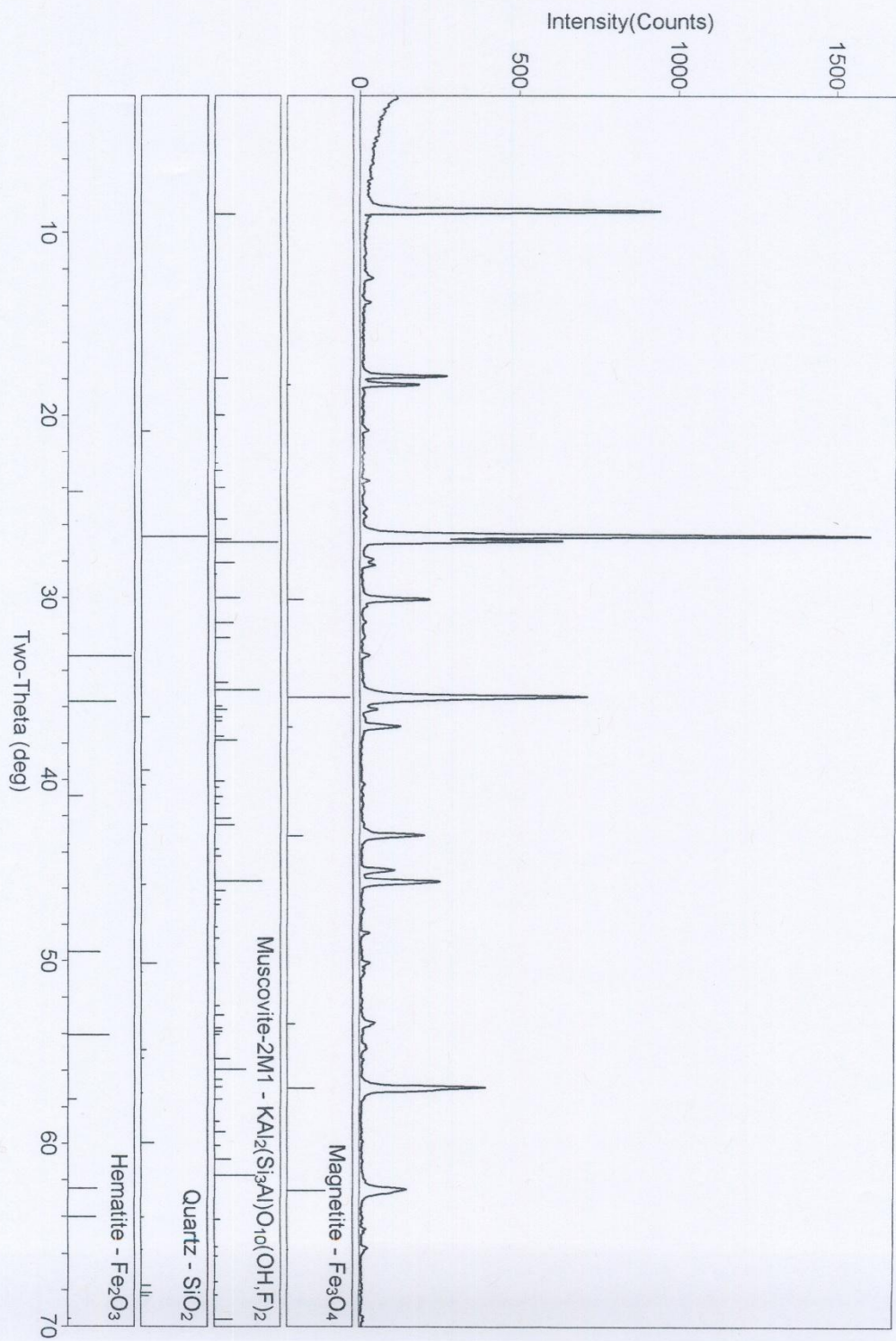SCAN: 2.5/70.0/0.02/0.6(sec), Cu(40kV,40mA), I(max)=1611, 06/09/09 03:21p
PROC: [New Quantitative Analysis]

Phase ID (3)	RIR	Wt%	Wt(n)%	Vol(n)%	#L	I%-I(r)	POC
☐ Magnetite - Fe_3O_4	5.13	17.2 (1.1)	17.2 (1.1)	10.0 (0.8)	5	3.6	<None>
☐ Muscovite-2M1 - $KAl_2(Si_3Al)O_{10}(OH,F)_2$	0.52	65.9 (4.2)	65.9 (4.2)	70.6 (5.7)	4	13.4	<None>
☐ Quartz - SiO_2	3.41	16.9 (1.1)	16.9 (1.1)	19.4 (1.6)	1	0.0	<None>

NOTE: Calculation Using: Peak Area, POC: Preferred Orientation Correction, Brindley Correction = 5.0 microns

2-Theta	FWHM	Height	H%	Area(a1)	A%	I(r)	I(p)	I%-I(r)	(h k l)
☐ 18.306 (0.013)	0.152 (0.012)	128 (10)	24.6	1093 (111)	14.3	5.1	5.1	9.2	(1 1 1)
☐ 30.076 (0.008)	0.166 (0.015)	166 (11)	31.9	1893 (154)	24.8	24.8	24.8	0.0	(2 2 0)
☐ 35.420 (0.003)	0.219 (0.005)	522 (8)	100.0	7629 (153)	100.0	100.0	100.0	0.0	(3 1 1)
☐ 37.043 (0.006)	0.187 (0.011)	97 (4)	18.6	1198 (66)	15.7	8.0	8.0	7.7	(2 2 2)
☐ 43.046 (0.003)	0.154 (0.010)	173 (7)	33.1	1995 (103)	26.2	24.8	24.8	1.4	(4 0 0)
☐ 8.864 (0.004)	0.217 (0.004)	650 (11)	100.0	7813 (176)	100.0	100.0	100.0	0.0	(0 0 2)
☐ 17.844 (0.010)	0.138 (0.010)	197 (15)	30.3	1712 (143)	21.9	20.0	20.0	1.9	(0 0 4)
☐ 20.805 (0.280)	0.199 (?)	14 (?)	2.1	201 (?)	2.6	3.0	3.0	-0.4	(1 1 1)
☐ 26.898 (0.006)	0.133 (0.007)	489 (24)	75.2	3806 (230)	48.7	100.0	100.0	-51.3	(0 0 6)
☐ 26.637 (0.003)	0.164 (0.005)	1180 (23)	100.0	11437 (364)	100.0	100.0	100.0	0.0	(1 0 1)

Intensity(Counts)

Two-Theta (deg)

Magnetite - Fe$_3$O$_4$

Muscovite-2M1 - KAl$_2$(Si$_3$Al)O$_{10}$(OH,F)$_2$

Quartz - SiO$_2$

Hematite - Fe$_2$O$_3$

Quantitative Analysis from Profile-Fitted Peaks

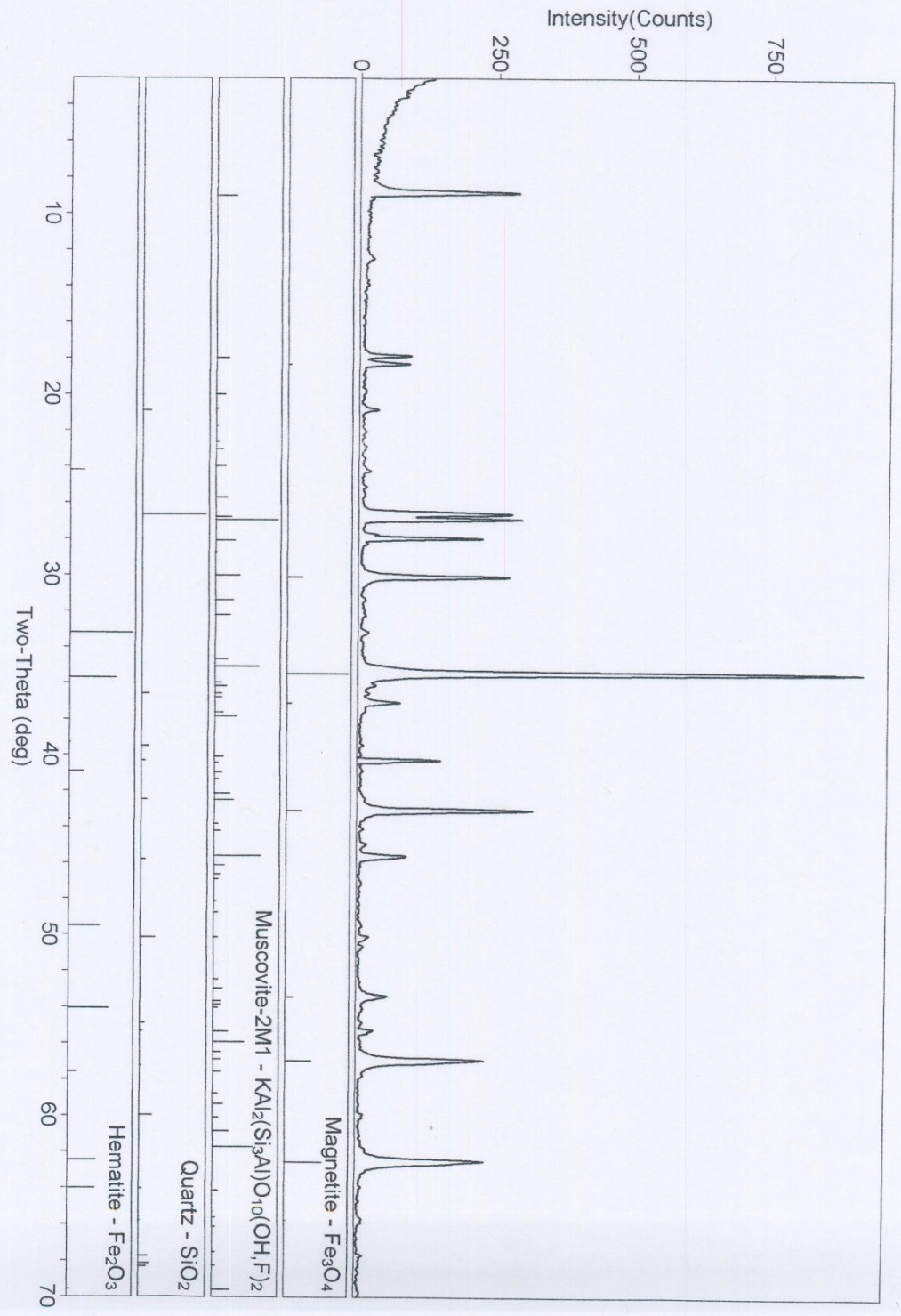FILE: [089-0445.raw] Drum
SCAN: 2.5/70.0/0.02/0.6(sec), Cu(40kV,40mA), I(max)=921, 06/09/09 02:45p
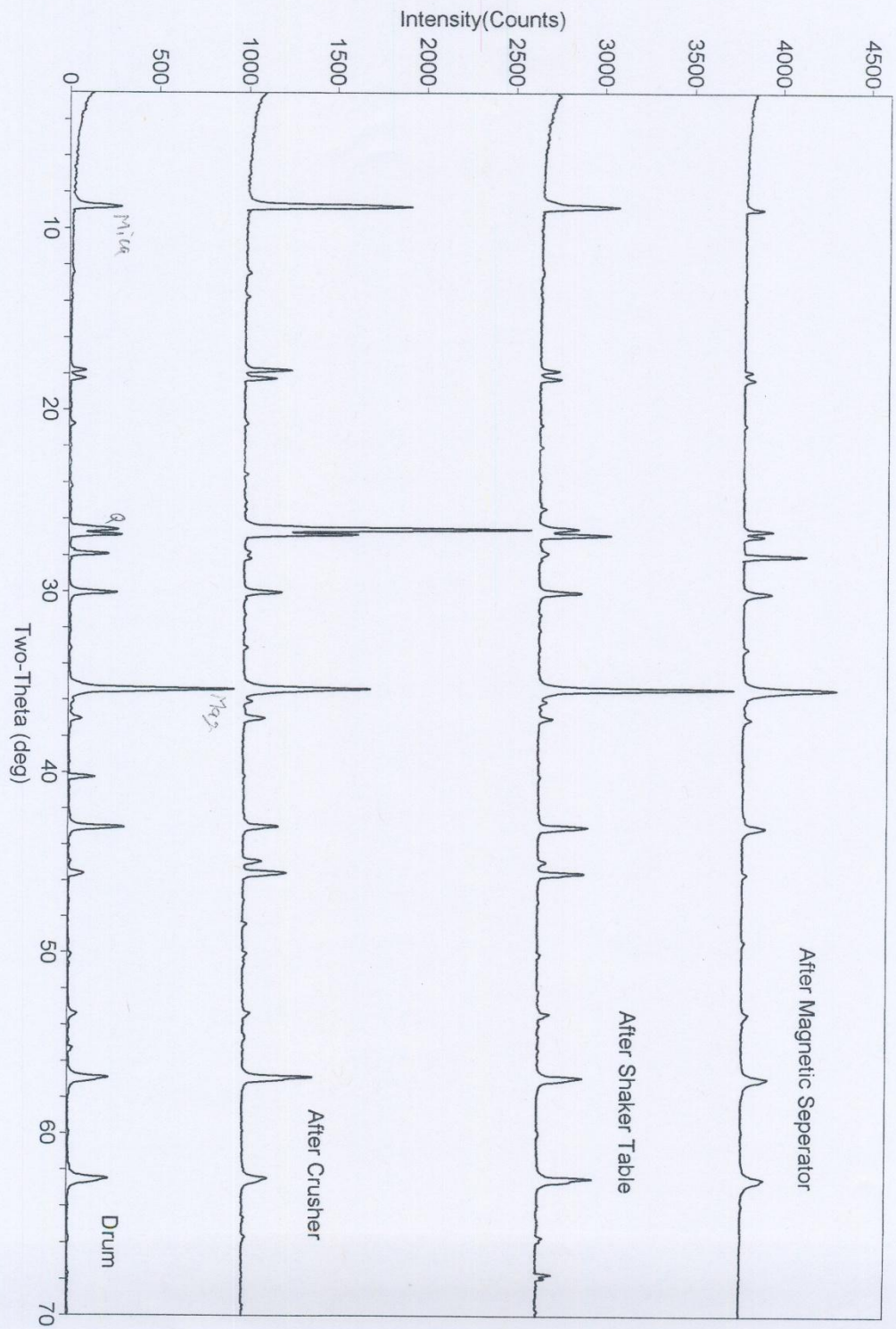PROC: [New Quantitative Analysis]

Phase ID (4)	RIR	Wt%	Wt(n)%	Vol(n)%	#L	I%-I(r)	POC
Magnetite - Fe_3O_4	5.13	30.4 (10.3)	30.4 (10.3)	19.2 (8.1)	6	6.3	<None>
Muscovite-2M1 - $KAl_2(Si_3Al)O_{10}(OH,F)_2$	0.52	62.1 (21.0)	62.1 (21.0)	71.8 (30.3)	4	15.3	<None>
Quartz - SiO_2	3.41	7.0 (2.4)	7.0 (2.4)	8.6 (3.6)	2	1.0	<None>
Hematite - Fe_2O_3	3.24	0.5 (0.2)	0.5 (0.2)	0.3 (0.1)	1	0.0	<None>

NOTE: Calculation Using: Peak Area, POC: Preferred Orientation Correction, Brindley Correction = 5.0 microns

2-Theta	FWHM	Height	H%	Area(a1)	A%	I(r)	I(p)	I%-I(r)	(h k l)
18.288 (0.012)	0.176 (0.017)	60 (4)	8.1	723 (62)	8.1	5.1	5.1	3.0	(1 1 1)
30.072 (0.004)	0.173 (0.007)	207 (6)	27.8	2458 (88)	27.5	24.8	24.8	2.7	(2 2 0)
35.429 (0.002)	0.170 (0.004)	743 (11)	100.0	8947 (166)	100.0	100.0	100.0	0.0	(3 1 1)
37.060 (0.007)	0.175 (0.018)	56 (3)	7.5	719 (60)	8.0	8.0	8.0	0.0	(2 2 2)
43.060 (0.006)	0.160 (0.015)	272 (18)	36.6	3057 (245)	34.2	24.8	24.8	9.4	(4 0 0)
62.525 (0.008)	0.232 (0.023)	202 (13)	27.2	3518 (278)	39.3	62.0	62.0	-22.7	(4 4 0)
8.883 (0.006)	0.236 (0.007)	181 (4)	80.2	2567 (84)	100.0	100.0	100.0	0.0	(0 0 2)
17.852 (0.010)	0.138 (0.013)	65 (5)	28.6	595 (57)	23.2	20.0	20.0	3.2	(0 0 4)
26.888 (0.009)	0.137 (0.016)	226 (15)	100.0	1751 (205)	68.2	100.0	100.0	-31.8	(0 0 6)
27.955 (0.003)	0.122 (0.006)	182 (7)	80.5	1467 (68)	57.1	31.0	31.0	26.1	(1 1 4)
20.824 (0.019)	0.163 (0.041)	22 (4)	11.2	266 (54)	10.9	13.0	13.0	-2.1	(1 0 0)
26.587 (0.015)	0.225 (0.011)	193 (8)	100.0	2441 (133)	100.0	100.0	100.0	0.0	(1 0 1)
33.143 (0.058)	0.217 (0.057)	9 (2)	100.0	106 (34)	100.0	100.0	100.0	0.0	(1 0 4)

X-ray diffraction pattern plotting Intensity (Counts) versus Two-Theta (deg), with reference patterns for Magnetite - Fe$_3$O$_4$, Muscovite-2M1 - KAl$_2$(Si$_3$Al)O$_{10}$(OH,F)$_2$, Quartz - SiO$_2$, and Hematite - Fe$_2$O$_3$.

Appendix C: Quantitative analysis and graphs for XRD for First Muffle
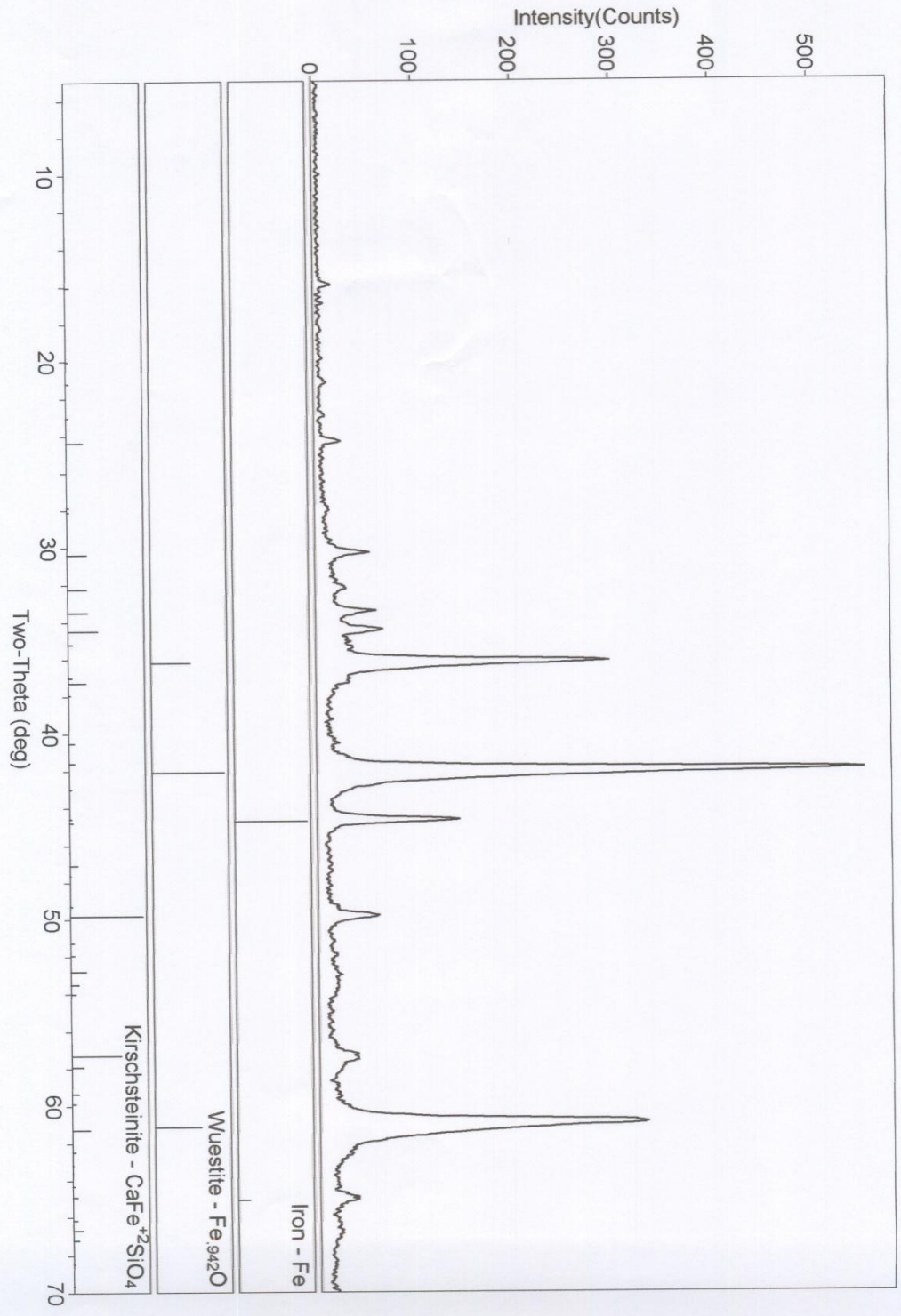Furnace test

Quantitative Analysis from Profile-Fitted Peaks

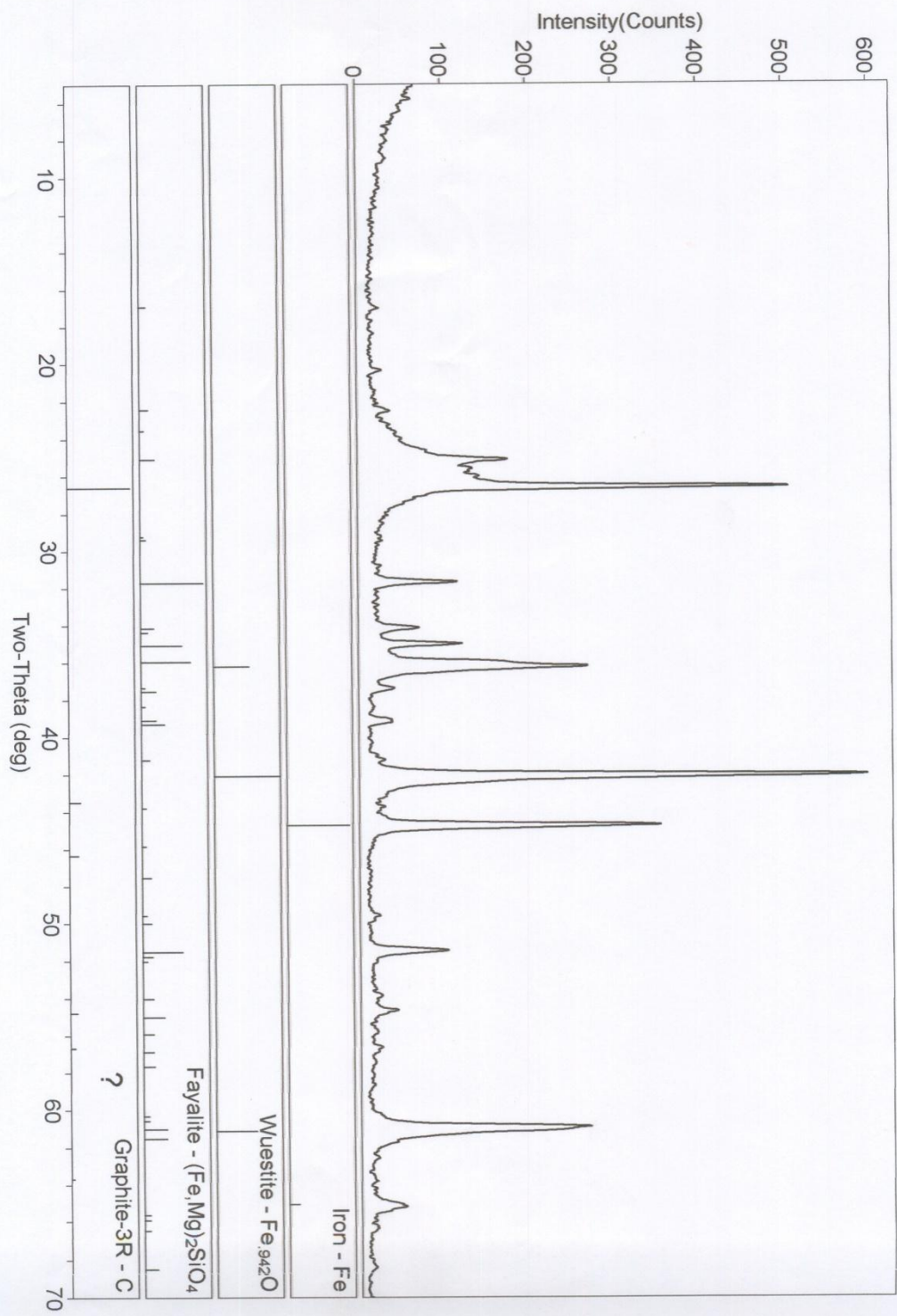FILE: [089-0453.raw] Cleveland Cliffs
SCAN: 5.0/70.0/0.02/0.6(sec), Cu(40kV,40mA), I(max)=552, 06/11/09 01:37p
PROC: [New Quantitative Analysis]

Phase ID (3)

■ Iron - Fe
■ Wuestite - $Fe_{.942}O$
■ Kirschsteinite - $CaFe^{+2}SiO_4$

	RIR	Wt%	Wt(n)%	Vol(n)%	#L%	I%-I(r)	POC
	10.77	6.4 (1.0)	6.4 (1.0)	4.3 (0.8)	1	0.0	<None>
	4.75	77.4 (11.9)	77.4 (11.9)	70.5 (13.5)	3	1.0	<None>
	1.25	16.2 (2.5)	16.2 (2.5)	25.2 (4.8)	1	0.0	<None>

NOTE: Calculation Using: Peak Area, POC: Preferred Orientation Correction, Brindley Correction = 5.0 microns

2-Theta	FWHM	Height	H%	Area(a1)	A%	I(r)	I(p)	I%-I(r)	(h k l)
44.669 (0.010)	0.325 (0.008)	87 (2)	100.0	1510 (53)	100.0	100.0	100.0	0.0	(1 1 0)
36.127 (0.009)	0.432 (0.013)	178 (3)	52.3	4722 (134)	42.4	54.4	54.4	-12.0	(1 1 1)
41.961 (0.006)	0.527 (0.008)	340 (3)	100.0	11136 (156)	100.0	100.0	100.0	0.0	(1 1 0)
60.843 (0.012)	0.675 (0.021)	214 (4)	63.0	9746 (264)	87.5	64.0	64.0	23.5	(1 2 1)
49.745 (0.034)	0.337 (0.064)	38 (4)	100.0	908 (135)	100.0	100.0	0.0	(0 4 2)

Intensity(Counts)

Two-Theta (deg)

Iron - Fe

Wuestite - Fe$_{.942}$O

Kirschsteinite - CaFe^{+2}SiO$_4$

Intensity(Counts)

600
500
400
300
200
100
0

Iron - Fe

Wuestite - Fe$_{.942}$O

Fayalite - (Fe,Mg)$_2$SiO$_4$

? Graphite-3R - C

10   20   30   40   50   60   70

Two-Theta (deg)

Homestake

44