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MAGNETIC CHARACTERIZATION OF IRON FORMATIONS FROM DRILL
CORES NEAR IRON MOUNTAIN, MICHIGAN

By

Adren Rigdon

A THESIS

Submitted in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

In Geophysics

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2019

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This thesis has been approved in partial fulfillment of the requirements for the Degree of MASTER OF SCIENCE in Geophysics.

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Abstract

The magnetic properties of the ~ 1.88 Ga Vulcan Iron Formation and Felch Formation were investigated from samples taken from three drill cores through the Felch Trough, north of Iron Mountain, MI. The conducted analyses included measurements of natural remanent magnetization, magnetic hysteresis, first order reversal curves, magnetic anisotropy, scanning electron microscopy, and electron dispersive spectroscopy. The data is compared to the results of a previous magnetic characterization study conducted on surface outcrop samples on two members of the Vulcan formation, the Curry and Traders members, near Iron Mountain, MI. Two distinct members of the Vulcan Formation were identified in the core samples that correlated to the members from the surface samples. While there were some similarities between the surface and core samples, the core samples presented higher magnetic susceptibilities by 1-2 orders of magnitude as well as a lower ratio of hematite to magnetite than the surface samples. The discrepancy may be due to the oxidation of magnetite to hematite by surface weathering. The obtained results, particularly the higher magnetic susceptibility, will be useful for interpretation of data collected by an aeromagnetic survey conducted in the area by the USGS.

Introduction

1.1 Background

Iron-rich sedimentary rocks in which iron oxide or iron sulfide minerals are interlayered or interspersed with silicates or carbonates are collectively known as iron formations (Gross, 1980). The nomenclature of iron bearing formations have varied over the years with various local definitions that have created some confusion when comparing iron formations on a global scale. Locally used terms for iron bearing formations included “iron-bearing formation” or “iron formation” in North America, “ironstone” or “banded hematite quartzite” in South Africa, “itabirite” in Brazil and Venezuela, and “jaspilite” to “banded iron-formation” in Australia (Trendall and Morris, 1983). Recently, iron formation or banded iron formations have become the more universal terms with a broad definition of “any sedimentary rock whose principal chemical characteristic is an anomalously high content of iron” (Trendall and Morris, 1983). James et al. (1961) suggested a very similar definition when defining iron formations in the Lake Superior region to be “a chemical sediment, typically thin-bedded or

laminated, containing 15 percent or more iron of sedimentary origin, commonly but not necessarily containing layers of chert” (James, 1954).

Similar to the nomenclature issues of iron formations, slightly different classification schemes for iron formations have been used. Based on their depositional regime and environment, Gross (1980) classified iron formations into four main types: Superior, Algoma, Minette, and Clinton (Gross, 1980). Trendall and Morris (1983), however, argued that the Minette and Clinton types were too subjective to be accepted for classification so that only two main types are currently used, the Algoma and Superior types (Trendall and Morris, 1983). The Algoma type formations are found in all ages of rock and are associated with greywacke sedimentary units and, most importantly, volcanicity, whereas the (Lake) Superior types are cherty and oolitic, being related to dolomites and black shales, and deposited on continental shelves near shoreline with little to no relation to volcanics (Gross, 1980).

Another classification between types of iron formations are banded iron formations (BIFs) and granular iron formations (GIFs), which is based on their texture. BIFs have a laminated or thinly bedded texture, whereas GIFs have no bedding or layering. The layering of the BIFs can have a wide variety of thicknesses with banding being on a scale of a meter to millimeters. The alternating layers of BIFs of the Superior type are typically represented by red and grey bands. The reddish bands are typically silicates such as jasper and garnet or chert but the reddish color can also indicate

a secondary oxidation of hematite. The grey bands are typically iron ores such as magnetite, hematite or iron silicates. One of the main theories for the difference between BIFs and GIFs is that while both were deposited in shallow seas, the GIFs were formed above the wave base and became agitated due to waves and storms preventing layers from forming (Klein, 2005).

BIFs have formed all over the world and over a wide range of time. From 3.8 Ga in West Greenland intermittently to 0.6 Ga in Western Australia at a maximum abundance towards the end of the Archean at 2.5 Ga (Klein, 2005). The large abundance of BIFs around 2.5 Ga are almost entirely Algoma type iron formations linked to the volcanic activity of orogenic environments that formed much of the late Archean greenstone belts (Trendall and Morris, 1983).

The most accepted depositional environment for iron formations is an anoxic water environment that can transport the dissolved iron to a place where oxidation can occur and cause the iron to precipitate. The theories about the mechanisms by which the iron could have been precipitated vary between authors and locations with some formations suggested to form from inorganic precipitation due to chemical mixing and some to form from biological processing (Klein, 2005). This mechanism is well understood for the Archean eon which extends from 4 Ga to 2.5 Ga before oxygen was prevalent in the atmosphere. The Great Oxidation Event (GOE) occurred around

2.4 Ga, which would cause a progressive oxidation of dissolved iron before being transported to a single location (Bekker et al., 2004).

In addition to the Archean IFs, there are two peaks of iron formation deposition that occur after the GOE, around 1.88 Ga and 0.8 Ga, when the atmospheric oxygen should have prevented these occurrences. The peak around 1.88 Ga has been linked to large scale submarine volcanism and crustal growth as the introduction of mafic-ultramafic lava flows overwhelmed areas of low oxygenated waters and allowed for the reemergence of iron formations (Rasmussen et al., 2012). The appearance of Neoproterozoic iron formation around 0.8 Ga has been suggested to have been caused by anoxic events from water bodies becoming capped with glaciers allowing the iron to accrete and then precipitate with oxygen from the atmosphere after the removal of the glaciers (Baldwin et al., 2012).

Iron formations may hold information that can tell us about the environmental conditions that were present at the time of their deposition. Depending on the conditions that the iron was precipitated from, inferences about early atmospheric conditions or early biological production of oxygen can be made. Investigations of Proterozoic iron formations is especially important as they may shed light on the depositional environments occurring after the GOE. Iron formations also hold magnetic information from the early period of Earth's history. Highly metamorphosed formations are difficult to read but natural remanent magnetization that was formed during deposition can

yield information about the magnetic field at the time of deposition. This data be used to understand how the Earth’s magnetic field was formed and evolved, including its orientation and magnitude.

Iron formations are also the largest source of iron and are heavily mined throughout the world. The Lake Superior region has one of the largest supplies of BIFs and from 1848 to 1983 had shipped 4.6 billion metric tons of iron ore. In 1994, 95% of the U.S. supply of iron ore was sourced from the Lake Superior region. Currently, the US is still a net exporter of steel which is produced using much of the iron ore sourced from the Lake Superior region (USGS, 2018). Exploration and economical assessment of the mineral resources using geophysical methods requires knowledge of the physical properties of iron formations. In particular, aeromagnetic survey represents the most efficient geophysical method for mapping iron formations to estimate their extent, structure and economic potential (e.g., Clark et al. (2015)). Proper geologic interpretation of the aeromagnetic data however requires detailed and independent information on the magnetic properties of rocks (magnetic petrophysics).

This project focuses on the Vulcan Iron Formation and the Felch ferruginous Formation in the Menominee Range in the vicinity of Iron Mountain, MI. The Menominee Group belongs to the Marquette Range Super Group that formed during the Penokean Orogeny, which occurred around 1.875-1.835 Ga and, more specifically, the Vulcan and Felch formations have been dated to $1,874 \pm 9$ Ma (Schneider et al., 2002).

The Vulcan Iron Formation is associated with strong magnetic anomalies observed in ground and airborne surveys (e.g., James et al. 1961, Bayley et al. 1966, Ben Drenth pers. comm.).

A previous graduate student at the department of GMES, Matthew Laird, conducted a rock magnetic, paleomagnetic, and scanning electron microscopy investigation of the samples of an upper (Curry) and a lower (Traders) member the Vulcan Iron Formation taken from surface outcrops (Laird, 2017). The obtained data led to a conclusion that a significant difference in magnetic properties between the two members existed with the main difference based on the hematite-to-magnetite ratio. The Curry member was designated as a granular iron formation dominated by hematite as opposed to the magnetite-dominated banded iron formation of the Traders member (Laird, 2017). However, the values of bulk magnetic susceptibility obtained from the surface outcrop samples were unable to explain strong magnetic anomalies acquired by a US Geological Survey's (USGS) aeromagnetic survey over the area (Ben Drenth, pers. comm.). One possible reason for this discrepancy is that the surface outcrops may be affected by weathering and thus may not represent the bulk magnetic properties of the studied iron formations.

To address this possibility, in this project, a detailed magnetic and scanning electron microscopy investigation of the Vulcan and Felch formations was conducted on samples taken from drill cores. These samples have not been affected by weathering.

The conducted experimental work includes measurements of the natural remanent magnetizations, magnetic hysteresis, bulk magnetic susceptibility and anisotropy, thermomagnetic analyses, scanning electron microscopy, and electron dispersive spectroscopy. The obtained magnetic characterization data will inform the on-going efforts of the USGS to generate a detailed aeromagnetic map of the eastern Upper Peninsula of Michigan that includes the Vulcan Formation. In addition, the Vulcan Iron Formation formed during the late Paleoproterozoic peak of iron formation development represents a great interest in terms of understanding the Earth system conditions at that time, including the mechanism of iron formation deposition. Overall, the obtained rock magnetic data contribute to an observational database that can assist better understanding the Precambrian Earth processes.

1.2 Geologic Settings

The area that is studied in this thesis is near Iron Mountain, Michigan near the border of Wisconsin as shown in figure 1.1. Three cores representing iron formations were identified from log records and acquired from the Department of Environmental Quality's Michigan Geologic Core and Sample Depository in Marquette, MI whose locations are shown in figure 1.2. The first is labeled G1001 and is located in the Felch trough of the Groveland Mine area. The depths of the core samples from G1001 range from 14 ft to 70 ft but it is the only core drilled at a 90° angle. Due to the observed

Figure 1.1: Overview of area in question

tight grey banding with well defined lamination, the G1001 cores most likely represent the lower member of the Vulcan Formation.

Figure 1.2: Drill core locations shown with yellow stars on top of a geologic map with the geological units shown on the right (Sims and Schulz, 1993).

The next core, labeled C64, is located just north of a different outcrop of the Vulcan formation (figure 1.2 and 1.3). The depths of these core samples are from 130 ft to 135 ft, which corresponds to the fault zone in figure 1.3(b). Since the core samples come from the fault zone it is difficult to relate the samples to a certain formation or member but there are some similarities. The non-existent banding and large iron inclusions within a silica sand matrix and not a crystallized quartz could be consistent with the quartz-mica facies of the Felch formation being weathered into a sandy hematitic structure before being within a fault zone (James et al., 1961).

(a)

(b)

Figure 1.3: a) A map which displays the zoomed in area of C64. The red lines are isomagnetic contour lines showing magnetic anomalies and the red numbers are dip-needle values. b) The cross section showing the depth and direction of the drill hole and the geologic formations encompassed (James et al., 1961).

The last location is a core labeled C27 which is located west of the C64 location and just north of a local outcrop of the Vulcan formation shown in figure 1.4(a). The drill core is directed south through two sets of the Vulcan Formation as shown in figure 1.4(b). These core samples represent two different segments of the Vulcan

Iron Formation with the first being at a depth of 70 ft to 135 ft. The gray and intermittent banding with some mosaic or light granular texture is consistent with the upper (Curry member) Vulcan formation (James et al. 1961, Laird 2017). The second formation from 230 ft to 242 ft is very well banded between layers of metachert and silvery gray iron bands characteristic of the lower (Traders member) Vulcan Formation (James et al. 1961, Laird 2017). This creates a difficult interpretation as the Michigamme Slate is the overlying formation on top of the Vulcan formation so an initial hypothesis would be that the Vulcan formation has been overturned and the deeper core should correlate to the upper (Curry member) formation.

(a)

(b)

Figure 1.4: a) A map which displays the zoomed in area of C27. The red lines are isomagnetic contour lines showing magnetic anomalies and the red numbers are dip-needle values. b) The cross section showing the depth and direction of the drill hole and the geologic formations encompassed (James et al., 1961).

The main iron bearing formation that this paper will address is the Vulcan Iron Formation with the samples from C64 possibly coming from the Felch Formation, underlying the Vulcan Formation within the Menominee Group. The Felch Formation is defined by its position between the Randville Dolomite and the Vulcan Iron Formation and the western edge extends through the Groveland mine area which is where site G1001 is located. The formation consists of many rock types including quartzite, quartz-mica schist, magnetite bearing quartzite and schists as well as a

unique grunerite-garnet-magnetic rock (James et al., 1961). The Vulcan Iron Formation covers a small area mostly confined within the Felch trough. Inside the Felch trough, the formation is generally a gray banded formation with some oolitic textures. The banding consists of a light gray saccharoidal metachert and magnetite bearing iron oxides and is approximately 0.1 to 0.3 inches in thickness (James et al., 1961). The Vulcan Formation in central Dickinson County has a maximum thickness of 400 ft (most likely 250 ft due to folding) which is much more eroded than where it appears in Southern Dickinson County where it consists of two members and is 600 ft thick. The Felch Formation is widely variable in thickness at 5 ft minimum to a few hundred ft maximum. The Felch Formation lays on top of the Randville Dolomite as an unconformity and the dolomite contains some small layers of shale with disseminated magnetite (James et al., 1961). From Gross (1980), the Superior type was based on iron formations in this area and are associated with dolomites and black shales which place the Felch and Vulcan Formations as typical Superior type iron formations.

The Felch trough where G1001 is located is a strong syncline which indicates at least some metamorphism occurred. While Trendall and Morris (1983), decided to not classify iron formations based on facies, James earlier classified the Vulcan Formation as an oxide facies containing thinly laminated hematite bands (James, 1954). The oxide facies indicates a large amount of oxygen that would cause the iron to precipitate quickly and is consistent with the oxygenated atmosphere.

The formation of the Menominee group which contains the Felch and Vulcan Formations took place at the time of the Penokean Orogeny around 1.88 Ga. The Penokean Orogeny occurred when an oceanic arc collided with the Archean Superior craton. This collision resulted in a foredeep on the South side of the collision which allowed for the iron formations to be deposited in wide shallow basins (Schulz and Cannon, 2007). Some of the deposits were not fully below wave base which resulted in less fine and distinct banding of the iron deposits. These foredeeps were most likely in restricted basins that prevented access to larger bodies of water or strong circulations which allowed for continuous banding due to even precipitation of the iron ores (James, 1954).

The samples that were taken for the previous magnetic characterization were acquired south of the locations of the drill sites (figure 1.5). As stated earlier, the distance as well as the folding and faulting between the surface and core samples will most likely result in some mineralogical and magnetic differences between the samples. There is no map detailing the separate Vulcan formations that are crossed by the drill cores so only visible and magnetic characterization produced throughout this thesis will be used to compare the core samples to the surface samples.

Figure 1.5: Drill cores used for this thesis shown in yellow stars to the north and the surface samples taken for previous analysis shown in blue stars to the south of the map (Sims and Schulz, 1993).

Methods

2.1 Sample Preparation

All of the investigated core samples were collected in the Department of Environmental Quality's Michigan Geologic Core and Sample Depository in Marquette, MI. Samples from three main sites were chosen. The first is from the Calumet Exploration of the Hanna Coal & Ore Corp at a site designated as C27. The drilling was started on June 18th of 1949 and was finished seven days later on June 25th. The hole was drilled 337 ft E. 390 ft S of N $\frac{1}{4}$ Corner of Section 15 Township 41 Range 29 in Michigan which is at 45.955711°N 87.928018°W in decimal degrees. The core has a diameter of approximately 3 cm and was drilled at a -30° angle in the direction of S14°E to a depth of 475 ft.

The second core is also from the Hanna Coal & Ore Corp in the Calumet District at a site designated C64. The hole was drilled from April 17th to May 1st of 1950 at an angle of -45° in the S27°E direction to a depth of 430 ft with an approximate core diameter of 5 cm. The site is located 1220'E, 900'S from N $\frac{1}{4}$ Corner of Section 8

Township 41 and Range 28 in Michigan which is 45.968475°N 87.841116°W in decimal degrees.

The last site is from the M. A. Hanna Company located at 2800 N 4000 E Section 4000 related to their companies coordinate system which is 45.9924°N 87.9822°W in decimal degrees. The site name is G1001 and was drilled 90° angle to 458 ft in April of 1968 with a core diameter of approximately 3 cm.

In order to preserve the core material for future research, half of the core sample that was taken must be returned to the repository. This meant that in some cases only quarter sections of the cores could be used to create the sample blocks for measurement purposes. A diamond blade saw in the Rock Preparation Lab at Michigan Technological University was used to split the cores in half to form quarters and in the case for site C64 a whole core was split in half. From these quarter and half cores all of the samples were prepared (figure 2.1).

Figure 2.1: Sample cores taken from the DEQ Michigan Geologic Core and Sample Depository for site C27 after being split in half.

Visual inspection showed that the samples from site C27 (figure 2.1) contained two different iron formations. The first iron formation in the core was observed from a depth of 68 ft to 142 ft and contains the samples taken at 70, 90, 108, 125, and 135 ft. The second iron formation is located between 227 to 250 ft and is separated from the first one by a layer of biotite schist located at 142 ft to 227 ft. The samples from the second iron formation come from a depth of 230, 240, and 242 ft. The younger iron formation, at shallower depths, is more granular with light banding showing up in the sample at 70 ft and some banding structure on the 135 ft sample as well. The core

log description indicates that there is some fine grain hematite in the iron formation. This formation is mostly composed of a light grey chert with some red sediments in the sample at 108 ft and 135 ft. The sample at 125 ft contains some fairly large quartz bodies and, similar to the samples at 90 and 108 ft, the iron formation is not in bands but is more granular and dispersed throughout the core.

The older formation samples from the depths at 230, 240 and 242 ft all have a distinct banding of the iron between chert and jasper (figure 2.1). Most of the iron bands are approximately 1 mm in width with some up to 4 mm in width. The spacing is varied with the light grey chert approximately 2 mm in width in the larger areas and in the very tight spots the width of the iron, jasper, and chert is less than 1 mm. The deepest sample at 242 ft has more jasper and an overall darker color than the lighter grey chert in the samples at 230 and 240 ft. The banding of the jasper and chert is around at 3-4 mm in width with some small 1 mm bands of iron at random intervals.

Figure 2.2: Sample cores taken from the DEQ Michigan Geologic Core and Sample Depository for site C64 after being split in half.

There are two samples from the site C64 (figure 2.2) and they come at a depth of 130 ft and 135 ft. The core description labels the depths from 119 to 140 ft as a mixture of hematite iron formation, breccia, silica sand matrix and some quartzite. The iron is not banded and is trapped in a loose matrix that is very crumbly and the red coloration leeches quickly when water is applied. The iron is in a few large clumps and is dispersed throughout the rock in smaller pieces.

Figure 2.3: Sample cores taken from the DEQ Michigan Geologic Core and Sample Depository for site G1001 after being split in half.

Five samples were taken at site G1001 at depths 14, 15, 43, 52, and 70 ft (figure 2.3). The iron banding is well defined and the bands vary in width from 1 mm up to 1.5 cm. The banding is often in the same direction as the core (90°) but is wavy and often contorted with small brecciated zones. The light grey chert is homogeneous with little coloration. Iron banding contains some fine grained hematite within larger crystals of magnetite. The bands in samples at 15 ft and 52 ft are the most contorted and wavy, while the bands at 70 ft are well defined. The samples at 14 ft and 43 ft have more of the brecciated zones within the iron banding, but not so much that it would resemble a granular formation.

Figure 2.4: G1001 sample from a depth of 14 feet with the cube that was cut from the quarter of the core.

Cubic specimens ($\sim 1 \times 1 \times 1$ cm) for paleomagnetic and bulk magnetic susceptibility measurements were first cut from the quarter/half cores using the diamond blade saw (figure 2.4). After cutting, the leftover pieces were placed in a titanium mortar and pestle to be crushed into small chips around 1 mm long and varying in width and height up to 1 mm exemplified in figure 2.5(a). These chips were taken from both the dark iron banding and the surrounding chert or red sediment to be used for magnetic hysteresis and first-order reversal curve (FORC) measurements. Several chips of the iron banding and the surrounding rock were then further powdered using the mortar and pestle for thermomagnetic analysis (figures 2.5(b) and 2.5(c)).

(a) Chip sample from site C27 at a depth of 125 ft and showing the darker bands of iron. (b) Powdered sample from site C27 at a depth of 125 ft showing the darker bands of iron. (c) Powdered sample from site C27 at a depth of 125 ft and showing the lighter colored chert.

Figure 2.5: Examples for the chip samples for magnetic hysteresis and FORC curves (a) as well as the powdered samples of the dark iron banding (b) and background chert (c) from the 125 ft sample from site C27.

The sample preparation for the FEI Philips XL 40 Environmental Scanning Electron Microscope (SEM) analysis started with cutting off pieces of the quarter core that were at least a centimeter or two long in each direction. It was important to make sure that the samples included the iron bands as well as a representative section of the background matrix so that all the lithologies present in the rock could be analyzed using one sample under the Scanning Electron Microscope. One sample for sites C64 and G1001 and two samples for site C27 were selected to check if there was a major difference in composition by depth which corresponded to the thermomagnetic analysis curves. These samples were placed in a mold and filled with epoxy which was then hardened (figure 2.6). The samples were then polished using increasingly smaller grit sizes starting with a 400 or 600 grit. After the initial manual polishing

using the large grit, the samples were polished using an automatic sample polisher with a 6 μm , 3 μm , and finally a 1 μm diamond grit paste to polish out all scratches. The last step was to coat the samples in carbon after which they were ready to be investigated with the SEM.

Figure 2.6: The SEM samples after being set in epoxy. One sample from sites C64 and G1001, and two samples from site C27.

2.2 Natural Remanent Magnetization (NRM)

The natural remanent magnetization (NRM) was measured using a 2G Enterprises 760-R Superconducting Rock Magnetometer (figure 2.7). First the data files had to be initialized with the core orientation information so that the software can correct for the position and angle of the sample to a normalized plot. The 2G Superconducting Rock Magnetometer is fit with a mylar tube that allows small cylindrical sections of a drill core to be inserted. In order to fit the cubic samples cut from the quarter cores, a sample holder adapter was used. The sample holder was measured while empty and, if a strong magnetization was observed, the holder was cleaned using 99% isopropyl alcohol and placed in an alternating field (AF) in order to remove the parasite magnetization. After the sample holder was cleaned and demagnetized, the cubes were placed in the holder and the NRMs of all the samples were measured.

Figure 2.7: A 2G Enterprises 760-R Superconducting Rock Magnetometer located in a magnetically shielded room in the Earth Magnetism lab of Michigan Technological University.

After measuring the NRM, the samples were placed in a liquid nitrogen bath inside of a magnetically shielded enclosure in order to remove viscous remanent magnetization components. This is achieved by cycling the specimens through the Verwey transition which occurs around 120 K and the magnetic anisotropy constant for magnetite reaches zero causing the multidomain grains to demagnetize and the shape dependent single domain grains to recover their initial magnetization before sitting in the Earth's magnetic field for long periods of time (McElhinny and McFadden, 2000).

An ASC TD-48SC Thermal Specimen Demagnetizer (figure 2.8(a)) was then used to demagnetize the cube specimens. This was accomplished by heating the samples to

a specific temperature in an argon atmosphere with the samples arranged on the furnace tray as shown in figure 2.8(b). As the specimens were slowly heated at specific temperatures, the minerals within the specimens would reach their Curie temperature and lose their magnetization. The remaining magnetization was measured and recorded after each heating step using the 2G magnetometer to identify the magnetization components. The heating steps started at 50-100°C until they were reduced around the Curie temperatures of magnetite, at 580°C, and hematite, at 680°C. The measurements were concluded when the magnetization fell below confidence levels, typically about 5% of the initial magnetization or 700°C, whichever occurred first.

Figure 2.8: (a) The ASC TD-48SC Thermal Specimen Demagnetizer used to heat the specimens for thermal stepwise demagnetization.(b) Sample holder containing the specimens that is inserted into the thermal demagnetizer.

2.3 Anisotropy of Magnetic Susceptibility (AMS) and Bulk Magnetic Susceptibility

Anisotropy of magnetic susceptibility (AMS) describes the differences in magnetic susceptibility measured along the three orthogonal principal axes. This is done by subjecting the sample to a magnetic field directed along the three axis and measuring the induced magnetization corresponding to each direction. An AGICO MFK1-FA Kappabridge was used in conjunction with the rotating holder (figure 2.9). The cubes for AMS must be separate from the cubes used for NRM, since the AMS measurements impart a magnetic field onto the sample which induces a magnetization of the samples that is different to the magnetization from their original location and will bias the NRM measurements. Similarly to the NRM measurements, the instrument holder is sized for a 1 inch diameter core and not a cube. In order to measure the small cubic specimens, they were placed in a plastic cube filled with cotton balls to ensure the specimen stayed in place and did not move during measurement.

Figure 2.9: AGICO MFK1-FA Kappabridge with the rotating holder for magnetic anisotropy.

The empty holder was first measured to ensure that it did not hold any magnetization to influence the samples. After calibrating and checking the holder, the cube is first placed with the positive z direction facing up and the inner core face that was cut facing outwards towards the front of the machine (figure 2.10(a)). The sample is then lowered into the Kappabridge where a field of 200 A/m is applied and the resulting magnetization is measured. This is repeated for the sample being rotated 90° so that, facing the machine, the inner core face is now looking to the right (figure 2.10(b))

and the last measurement is taken with the positive z pointing toward the front of the machine and the inner core face looking downwards into the machine depicted in figure 2.10(c). The three measurements are used to find the magnetic susceptibility in each of the directions and record the values as $K1$, $K2$, and $K3$, with $K1$ being the largest susceptibility and $K3$ being the smallest. After the three directions are measured, the bulk magnetic susceptibility is measured which does not depend on the samples orientation.

(a) Anisotropy position 1 with the z direction facing towards the front.

(b) Anisotropy position 2 with the z direction facing towards the right.

(c) Anisotropy position 3 with the z direction facing down and pointing towards the front.

Figure 2.10: Sample orientations for measuring the anisotropy of magnetic susceptibility.

2.4 Thermomagnetic Measurements

The powdered samples were weighed and then measured using the MFK1-FA Kappabridge with a CS-L and a CS-3 Furnace Apparatus shown in figure 2.11. The first

step was a low temperature run with the cryostat. The powder was cooled to approximately -185°C using liquid nitrogen and the magnetic susceptibility was measured as the powder was slowly returned to room temperature. The next step was the high temperature run using the CS-3 Furnace Apparatus where the sample was surrounded by argon gas and heated to approximately 700°C . Magnetic susceptibility was measured as the sample was heated and subsequently cooled back to room temperature. A final low temperature run using the liquid nitrogen bath and the cryostat finished the thermomagnetic measurements with each sample.

Figure 2.11: AGICO MFK1-FA Kappabridge with the CS-3 Furnace Apparatus.

The powdered samples were approximately 0.2 - 0.5 grams and as many as possible were split into separate specimens of the dark iron bands, the surrounding chert, or the red sediments. For the low temperature runs, the powder was poured into a quartz tube and a thermocouple was inserted so that the powder surrounded the thermocouple inside of the tube. The tube was then placed within the cryostat and liquid nitrogen was poured around the tube until the thermocouple registered a temperature reading below 188 °C. The remaining liquid nitrogen was expelled using argon gas and the magnetic susceptibility was measured about every 3°C increase until the thermocouple registered 0°C.

For the high temperature runs, the quartz tube containing the powdered sample and thermocouple were inserted into the CS-3 Furnace Apparatus. A tube with argon gas continuously running was then connected to the furnace apparatus and the measurements were automatically recorded for every 5°C increase in temperature until 700°C when it was the recorded for every 5°C decrease until the sample cooled back down to approximately room temperature. After the high temperature run, another low temperature run with the cryostat was conducted with the same method to complete the thermomagnetic curves.

After the thermomagnetic measurements, the data was processed using software named Cureval obtained from AGICO for use with the Kappabridge (Chadima and Hrouda, 2012). The processing allows for the curves to be inspected for inflection

points that will point to the Curie temperatures for different minerals. The Verwey transition occurs at approximately -153°C for magnetite (Verwey, 1939) and a similar transition for hematite that occurs at approximately -23°C is called the Morin transition (Morin, 1950).

2.5 Magnetic Hysteresis and First Order Reversal Curves (FORCs)

Magnetic hysteresis is important for characterizing the magnetic domain state and magnetic interactions between ferromagnetic minerals of the samples. An Alternating Gradient Magnetometer (AGM) was used to measure the magnetic hysteresis loops. Small rock chips (figure 2.5(a)) are attached to a probe (figure 2.13) using a silicone grease that is diamagnetic. This probe is then inserted into the AGM (figure 2.12) so that the rock chip is placed between two magnetic poles. These poles are used to create an alternating magnetic field which induces a vibration in the sample. The amplitude of the vibrations is proportional to the strength of the magnetic moment of the sample.

Figure 2.12: MicroMag Model 2900 Alternating Gradient Magnetometer at the Michigan Technological University's Earth Magnetism Lab.

Before the measurements, the AGM must be turned on for 15-30 min so that the instrument can become stable. After the machine has been allowed to stabilize, the first measurement is with a standard that has a magnetic moment of $77.64 \mu\text{emu}$ which is attached to the probe using a silicone grease that is diamagnetic. Before measuring the constant, the amplitude is calculated proportional to the sample's magnetic moment and this is done before each new measurement. This known constant is then used to calibrate the AGM with a magnetic hysteresis loop to ensure the current measurements will be consistent with previous and future measurements. After the

calibration is finished, a magnetic hysteresis loop is measured using the empty probe with a small dab of the silicone grease. The measured response should result in an inverse linear relationship between the magnetic moment (M) and the applied field (H). This magnetic moment response from the silicone grease will be subtracted from the future measurements. The diamagnetic silicone grease should result in a diagonal line from the bottom right corner to the top left corner and back to the bottom right corner to make one solid diagonal line without deviations on the M versus H plot. This calibration routine can sometimes take over an hour but is very important to ensure accurate measurements of the samples. The empty probe signal is then subtracted from the current hysteresis loop. Next, a correction to remove additional diamagnetic or paramagnetic signals was applied and the final result is saved as the adjusted hysteresis loop shown in figure 2.14(a).

Figure 2.13: A P3 Parallel Polymide Probe used for AGM measurements.

After the hysteresis loops are measured a DC curve (or backfield curve) is measured (figure 2.14(b)). For this, the sample is first placed in the maximum magnetic field that was used for hysteresis to induce the saturating magnetization (M_s). After the sample is saturated, the field is removed and the saturation magnetic remanence (M_r) is measured. Next, a small field in the opposite direction to the initial saturating field is applied in order to demagnetize the sample by a small amount. That small field is then turned off and, in the absence of any applied field, the isothermal magnetic remanence of the sample is measured. This is done until the remanence of the sample in the absence of a field reaches zero which is a point known as the coercivity of remanence (H_{cr}). The coercive force (H_c) measured during hysteresis is measured in the presence of a backfield whereas the coercivity of remanence is measured in the

absence of a backfield. A Day plot was devised to display the hysteresis parameters of multiple samples on one plot that also suggests whether the samples are single, pseudo-single, or multi domain by plotting M_s/M_r versus H_{cr}/H_c (Day et al., 1977).

Figure 2.14: a) A hysteresis loop for the sample at site C27 at a depth of 108 ft for the dark iron banding. The plot is annotated to show the important hysteresis parameters of magnetic saturation (M_s), magnetic remanence (M_r), and coercive force (H_c). b) A dc (backfield curve) for the same sample at site C27 at 108 ft depth for the dark iron banding. The maximum of magnetic remanence (M_r) and the coercivity of remanence (H_{cr}) is shown.

After the DC curves, the final measurement using the AGM was measurement of First Order Reversal Curves (FORCs), as constructed by Mayergoyz (1986), for each sample. FORCs are a composite of partial hysteresis curves all plotted on the same graph. The sample is placed in a saturating field which is then decreased to a low magnitude backfield, H_a . The partial hysteresis curve is recorded from H_a as the magnetic field is increased back to the saturating field. This partial curve is one first order reversal curve. Many of these curves are measured for an ever decreasing H_a until H_a becomes a saturating backfield. Points along the FORC are denoted as

$M(H_a, H_b)$ with H_a being the point where the magnitude of the backfield is the largest, where the curve begins, and H_b being the points along the curve starting at H_a till the saturating field is reached. The FORC curves are then processed using FORCinel v.3.0 software to form a diagram so that more information can be obtained from the image (Harrison and Feinberg, 2008). The diagram consists of a second derivative of the points as defined by

$$\rho(H_a, H_b) = (\partial^2 M(H_a, H_b)) / (\partial H_a \partial H_b) \quad (2.1)$$

where ρ is well defined for $H_b > H_a$ (Roberts et al., 2000). In order to make the diagram easier to read a rotation of 45° is applied by converting H_a and H_b to H_u and H_c as defined by

$$H_u = (H_a + H_b)/2 \quad (2.2)$$

$$H_c = (H_b - H_a)/2 \quad (2.3)$$

with H_c always being positive and plotted on the horizontal the graph becomes confined to the right plane (Roberts et al., 2000). A smoothing factor that determines the size of the local area to which the second order derivative is applied in order to reduce noise should generally be restricted to a factor of 2 to 5 where the size of the area is defined by $(2 * SF + 1)^2$ (Muxworthy and Roberts, 2007).

2.6 Königsberger Ratio

The Königsberger ratio is the ratio of the natural remanent component (J_{NRM}) to the induced remanent magnetization (J_i). J_{NRM} is found from the first measurement of the samples using the 2G Superconducting Magnetometer. J_i is found from the following equation:

$$J_i = \kappa H \quad (2.4)$$

The bulk susceptibility (κ) is found using the kappabridge during the anisotropic measurements while the ambient magnetic field intensity, H , was acquired using the International Geomagnetic Reference Field (IGRF) (IGRF, 2019). This allows for the Königsberger ratio to be calculated by

$$Q = \frac{J_{nrm}}{J_i} = \frac{J_{nrm}}{(\kappa H)} \quad (2.5)$$

For $Q \gg 1$ demonstrates a sample dominated by the NRM component where as $Q \ll 1$ demonstrates a sample dominated by induced magnetization. Also, a ratio of $Q \approx 1$ indicates a complex relationship between NRM and induced magnetization.

2.7 Scanning Electron Microscopy (SEM)

Figure 2.15: SEM samples being loaded into the Scanning Electron Microscope.

The SEM samples were placed to the loading tray of the FEI Philips XL 40 Environmental Scanning Electron Microscope (ESEM) as shown in figure 2.15. Once the samples were loaded, the tray was closed and the SEM was pumped into a vacuum. After the chamber had reached a vacuum of at least 10^{-4} Pascals, the electron beam was turned on and calibrated. An accelerating voltage of 15 kV was used to obtain electron dispersive microscopy (EDS) data, while a voltage of 5 kV was used to obtain secondary electron images of the samples. On backscatter electron (BSE) images

the iron bands showed brighter than the surrounding chert or sediment, and electron dispersive spectra were taken for both areas to characterize the composition for each lithology. Some EDS maps were taken to show the makeup of the iron bands and heavy silicate areas that surrounded them. Some rare earth elements were found and analyzed within the samples as well.

Results

3.1 Samples

Site C27 has some samples with well defined bands at depths of 70 ft and again at 230 ft, 240 ft, and 242 ft. The samples at 90 ft, 108 ft, 125 ft, and 135 ft are poorly banded and broken into small rectangular structures (figure 3.1). There are some red pockets at 108 ft and 135 ft before showing as thin or narrow bands in the deeper set of 230 ft to 242 ft. The red pockets are more similar to a hematite and silica matrix than a jasper chert. The sample at 135 ft has some yellow coloring which could be limonite that may have replaced some grunerite in a supergene process. The sample at 125 ft contains some large quartz crystals, whereas the other samples have lesser amounts of quartz. The deeper samples at 230 ft, 240 ft, and 242 ft are very well banded with chert, iron oxide, and hematite layers with very thin hematite (red) layers and thicker layers of magnetite and chert. The specimens were split into separate samples with the following naming convention: the specimens of the iron bands were labelled “Di”, the lighter silica chert specimens were labelled “Li”, and , for some of the

Figure 3.1: Samples from site C27 showing the cut faces.

deeper samples that could not be separated into well-defined lithology specimens, the specimens were labelled “Mi” (figure 3.17).

The samples for site C64 have the most unusual textures for the investigated area. Large iron inclusions are emplaced within a silica sand matrix. Some of the iron inclusions are large and rectangular, while there are also some smaller oolitic type inclusions within the silica sand (figure 3.2). When cutting the samples, the red coloration of the silica sand washes out which indicates the presence of hematite. This makes it difficult to observe hematite under the SEM as most of the SEM specimen polishing is done with water, which causes most of the hematite in the silica matrix to be washed away. For some of the analyses, the samples were separated into the

iron and the red matrix specimens. The naming convention was to label the dark iron specimens “Di”, and the red silica sand matrix specimens “Ri”.

Figure 3.2: Samples from site C64 showing the large rectangular iron inclusions after a straight cut with a diamond saw.

The samples for sites at G1001 are very well banded, consisting almost entirely of the chert, quartz and magnetite banded layers with some grains of hematite interspersed (figure 3.3). The banding on these samples are mostly parallel with the core at a 90°. This sample was taken in the middle of the Felch Trough near the Groveland mine (figure 1.2) and the area is a syncline, which suggests movement and possible metamorphism. The naming for this site was the same as for C27: the darker iron banding was labelled “Di”, and the lighter background chert labelled “Li”.

Figure 3.3: Samples from site G1001 showing the flat cut side.

3.2 Natural Remanent Magnetization

The measurements of natural remanent magnetization (NRM) were taken on 15 samples in total, with 8 samples from site C27, 2 samples from site C64, and 5 samples from site G1001. Each site was drilled with different specifications (dip angle), and the G1001 was the only one that was drilled vertically at a 90° angle. The site at C27 was drilled at a 30 degree angle, and the site at C64 was drilled at an angle of 45° from vertical.

The NRM for the site C27 had a mean of $1.92 \pm 3.79 A/m$ with a range from 0.0129 to 10.7 A/m with a median of 0.113 A/m . The NRM for site C64 had a mean of $0.230 \pm 0.292 A/m$ with values of 0.437 and 0.0233 A/m . The NRM for G1001 had a mean of $10.3 \pm 4.60 A/m$ with a range from 2.89 to 14.2 A/m and a median of 12.7 A/m . The NRM ranges vary pretty strongly with the samples from G1001 being the strongest and most of the samples from C27 being the weakest.

Samples	$J_{NRM}(A/M)$	Declination ($^{\circ}$)	Inclination ($^{\circ}$)	# of steps
C27_70	1.34E+02	93.2	83.5	11
C27_90	5.03E+01	306.6	64.1	18
C27_125	6.89E-01	318.7	46.4	20
C27_135	1.62E-01	24	28.1	20
C27_230	8.80E-01	182.6	59.7	16
C27_240	2.01E-01	251.9	-57.6	11
C27_242	1.95E+00	343	43.6	11
Mean	2.69E+01	193.5	36.4	15.875
C64_130	5.49E+00	336.3	35.2	20
C64_135	2.93E-01	293.4	38.4	20
Mean	2.89E+00	314.9	36.8	20
G1001_14	1.79E+02	223.8	-61.2	11
G1001_15	1.60E+02	260.3	20.3	11
G1001_43	1.62E+02	297.4	-64.6	11
G1001_52	1.11E+02	109.5	-49.7	12
G1001_70	3.63E+00	323	-47.6	11
Mean	1.23E+02	242.8	-40.6	11.2

Table 3.1

Summary showing the volume corrected natural remanent magnetization component (J_{NRM} , declination and inclination in degrees and corrected for structural tilt of the samples for all sites, and the number of heating steps before they lost their magnetization or reached 700°C.

The first step after measuring the NRM, was to perform a low-temperature demagnetization (LTD) on the samples to remove the secondary NRM (Figure 3.4). Samples

at site C27 exhibited a wide range of demagnetization after the LTD step, from 15% to 75%. The shallow samples generally lost a smaller portion of the initial NRM, except for the shallowest sample at 70 ft that lost nearly 75% of the NRM. The samples from 90 ft to 135 ft lost 15% to 35% of the magnetization, whereas the samples at 230 ft to 242 ft lost 62% to 78% of the initial magnetization. Samples at site C64 were demagnetized to 48% and 60%. Four of five samples for G1001 lost 61% to 91% of their initial magnetization and one sample at 15 ft underwent a 32% loss.

Figure 3.4: Bar plot that shows the resulting demagnetization of the samples after a low-temperature demagnetization (LTD) process.

After the LTD, the samples were heated to 600°C in a sequence of temperature steps, and the resulting percentage of NRM is shown in figure 3.5. Four of the samples from C27 lost more than 95% of the original NRM which indicates the samples were magnetite-rich and passed the Curie temperature for magnetite (585°C), which includes the first sample at 70 ft and the deeper samples from 230 ft to 242 ft. The sample at 90 ft lost just above 91% of NRM, indicating that it is similarly rich in magnetite while the other samples at 108 ft to 135 ft range from 25% to 70% loss. The sample at 130 ft depth from site C64 lost 98% of the original NRM whereas the second sample at 135 ft only lost 65%. The sample retaining more than 80% of the original NRM indicates a mineral with a Curie temperature greater than 600°C such as hematite ($T_c = 685^\circ\text{C}$). All the samples from site G1001 lost 99% of their original NRM indicating the dominance of magnetite as a magnetic remanence carrier.

Figure 3.5: Bar plot showing the result of heating the samples to 600°C as a percentage of their original NRM.

Samples from 70, 90 and 125 ft from site C27 show a characteristic remanent magnetization (ChRM) that plots south to southwest with upward inclination (figure 3.7(a)). The sample at 90 ft (figure 3.6(a)) presents a single southwest and down component gradually decreasing to the origin. The sample at 242 ft (figure 3.6(b)) presents an original northwest and down component that becomes a north and down component after LTD. Samples from 230 ft to 240 ft show a ChRM of north to northwest with upward inclinations (figure 3.7(b)).

Figure 3.6: Vector endpoint diagrams for samples at site C27 a) 90ft b) 242 ft. Red squares are data point projections on the horizontal plane while the blue squares are data point projections on the vertical plane. The first squares indicate the NRM component and subsequent changes in components are annotated with the temperature that the sample was heated to or LTD for low temperature demagnetization.

Figure 3.7: Equal Area plots for samples at site C27 a) 90 ft b) 242 ft.

C64 had two samples and the first at 130ft (figure 3.8(a)) displays a single component at southwest pointing up that loses almost half of its strength after the initial LTD. The second sample at 135 ft (figure 3.8(b)) originally displays a northwest and a horizontal inclination that gradually moves to a northwest and upward component until 685°C. The major declinations is shown in the equal area plots in figure 3.9.

Figure 3.8: Vector endpoint diagrams for samples at site C64 a) 130 ft b) 135 ft. Red squares are data point projections on the horizontal plane while the blue squares are data point projections on the vertical plane. Changes in components are annotated with the temperature that the sample was heated to or LTD for low temperature demagnetization.

Figure 3.9: Equal Area plots for samples at site C64 a) 130 ft b) 135 ft.

Two of the samples from site G1001 manifested a well-defined single low-coercivity/temperature NRM component (figure 3.10). The samples for G1001 vary with mostly centered to pointing north with downward inclinations as shown in figure 3.11(b).

The samples for G1001 present varying declinations with an overall trend of an upward inclination consistent with synclinal formation at the location of the drill core (figure 1.2). The sample at 14 ft (figure 3.10(a)) has an initial NRM component pointing southwest and down before moving to a low coercivity component pointing west and slightly up after the LTD. The sample at 70 ft (figure 3.10(b)) shows a single component that is northwest and upward that drops significantly after the LTD and drops significantly again at $\sim 580^{\circ}\text{C}$ to the origin. The other samples show declinations at northeast but with upward inclinations and a low coercivity all indicative of magnetite bearing minerals.

Figure 3.10: Vector endpoint diagrams for samples at site G1001 a) 14 ft b) 70 ft. Red squares are data point projections on the horizontal plane while the blue squares are data point projections on the vertical plane. Changes in components are annotated with the temperature that the sample was heated to or LTD for low temperature demagnetization.

Figure 3.11: Equal area plots for samples at site G1001 a) 14 ft b) 70 ft.

3.3 Thermomagnetic Analysis of Magnetic Susceptibility

Thermomagnetic analysis was conducted on 13 samples for site C27. One sample of the iron banding and one sample of the silicate matrix were analyzed for each of the cores from depths 70ft to 135ft (Figures 3.12-3.16). Only one sample that contained a mixture of the iron bands and the silicate chert were analyzed for samples 230ft to 242ft (Figure 3.17). All the measured samples exhibit an inflection point around 585°C which is consistent for a Curie temperature of magnetite. Eight of the plots indicate the presence of a ferromagnetic mineral phase with the Curie temperature around 670-680°C, most likely hematite (Figures 3.12-3.15).

The samples for the silicate (Li) at 90ft and all samples from 108ft to 135ft show Verwey transition shifted slightly colder than -150°C indicative of a small degree of oxidation. The plots from 90ft, 108ft and 135ft show little to no Verwey transition during the initial low temperature heating phase but do have a Verwey transition during the second low temperature phase that is consistent with magnetite (Figures 3.12, 3.13, 3.15). The initial lack of the Verwey transition that appears later indicates the creation of a pseudo-single-domain magnetite from a multi-domain grain mineral as multi-domain grains lack the Hopkinson effect during phase transitions (Dunlop,

2014). The samples at 90ft show a small inflection point on the heating curve around 360 to 370°C which does not occur in the cooling curve that could be due to the creation of the new magnetite (Figure 3.12).

These plots are irreversible and show small amounts of hematite being removed during the heating phase as the cooling phase of the high temperature indicates no hematite. Samples at 90 ft, 108 ft, and 135 ft also show a stronger Hopkinson peak, especially during the heating curve which is indicative of a single to pseudo-single-domain grain (Dunlop, 1974). A lower temperature inflection is observed on the cooling curve around 150 to 200°C which is most likely due to the formation of magnetite during the heating phase. Most of the plots that contain the low temperature inflection point contain evidence of hematite but are also nearly reversible from 600 to 700 °C which means the newly formed magnetite is most likely formed from the alteration of clays and not from hematite.

Figure 3.12: Thermomagnetic susceptibility plot that shows the dependence of low-field magnetic susceptibility versus temperature for sample at site C27 at 90ft a) iron banding and b) chert. The red line shows the first low-temperature run and the heating leg of the high-temperature run. The blue line shows the cooling leg of the high-temperature run and the post-heating low-temperature run.

Figure 3.13: Thermomagnetic susceptibility plot that shows the dependence of low-field magnetic susceptibility versus temperature for sample at site C27 at 108ft a) iron banding and b) chert. The red line shows the first low-temperature run and the heating leg of the high-temperature run. The blue line shows the cooling leg of the high-temperature run and the post-heating low-temperature run.

Figure 3.14: Thermomagnetic susceptibility plot that shows the dependence of low-field magnetic susceptibility versus temperature for sample at site C27 at 125ft a) iron banding and b) chert. The red line shows the first low-temperature run and the heating leg of the high-temperature run. The blue line shows the cooling leg of the high-temperature run and the post-heating low-temperature run.

Figure 3.15: Thermomagnetic susceptibility plot that shows the dependence of low-field magnetic susceptibility versus temperature for sample at site C27 at 135ft a) iron banding and b) chert. The red line shows the first low-temperature run and the heating leg of the high-temperature run. The blue line shows the cooling leg of the high-temperature run and the post-heating low-temperature run.

The other samples, 70 ft and 230 ft to 242 ft, have a well-expressed peak around -150°C on the first low-temperature curve (Figures 3.16, 3.17), consistent with the presence of stoichiometric magnetite. These curves are more reversible than the other curves from this site, showing only a slight increase in susceptibility after heating. No high-temperature mineral phase was observed above 600°C suggestion no or negligible amount of hematite. The weakly-expressed Hopkinson peaks at around 590°C indicate multidomain magnetite as the dominant magnetic mineral phase in these samples.

Figure 3.16: Thermomagnetic susceptibility plot that shows the dependence of low-field magnetic susceptibility versus temperature for sample at site C27 at 70ft a) iron banding and b) chert. The red line shows the first low-temperature run and the heating leg of the high-temperature run. The blue line shows the cooling leg of the high-temperature run and the post-heating low-temperature run.

Figure 3.17: Thermomagnetic susceptibility plot that shows the dependence of low-field magnetic susceptibility versus temperature for sample at site C27 a) 230ft, b) 240ft and c) 242ft. The red line shows the first low-temperature run and the heating leg of the high-temperature run. The blue line shows the cooling leg of the high-temperature run and the post-heating low-temperature run.

Four samples were used for thermomagnetic analysis of site C64 (Figure 3.18). The curves measured from these samples are similar and non-reversible. However, the two iron ("Di") samples show a higher magnitude of susceptibility than the silica matrix samples. All the samples exhibit the Curie temperature transitions around the 585°C indicating the presence of magnetite which is further supported by the Verwey transition peaks at -150°C on the first low-temperature curves. In addition, all the samples show an irreversible signal between 600°C and 700°C indicating the presence of hematite. The hematite signal is not seen on the cooling leg. A substantial increase in susceptibility upon cooling below 585°C and much stronger magnitudes of the Verwey transition for the second low-temperature run indicated that hematite is reduced to magnetite by heating. Interestingly, during cooling from 700°C, sample C64 at 130ft for the silica matrix shows a second Hopkinson peak at around 550°C in addition to the related to magnetite. This may indicate a bimodal range in the composition with the newly formed magnetite appearing to shift the hopkinson peak to a slightly cooler temperature (figure 3.18(b)).

Ten samples were analyzed for site G1001 (Figures 3.19-3.23). The samples are all relatively similar showing mostly reversible curves with a very small increase in susceptibility upon the high temperature cycling. The curves show very high magnetic susceptibilities for the iron samples while the chert samples have much lower susceptibility values compared to the iron sample from the same depth. All the samples reveal the presence of magnetite as indicated by the Curie temperatures near 585°C

Figure 3.18: Thermomagnetic susceptibility plot that shows the dependence of low-field magnetic susceptibility versus temperature for sample at site C64 at a) 130ft iron banding, b) 130ft red silica matrix, c) 135ft iron banding and d) 135ft red silica matrix. The red line shows the first low-temperature run and the heating leg of the high-temperature run. The blue line shows the cooling leg of the high-temperature run and the post-heating low-temperature run.

and by strong Verwey transitions. Very little to no hematite is present in any of the curves. The samples from the lowest depth at 70ft (figure 3.23) have a slightly less well-expressed Verwey transition at -150°C(Figure 3.23).

Figure 3.19: Thermomagnetic susceptibility plot that shows the dependence of low-field magnetic susceptibility versus temperature for sample at site G1001 at 14ft a) iron banding and b) chert. The red line shows the first low-temperature run and the heating leg of the high-temperature run. The blue line shows the cooling leg of the high-temperature run and the post-heating low-temperature run.

Figure 3.20: Thermomagnetic susceptibility plot that shows the dependence of low-field magnetic susceptibility versus temperature for sample at site G1001 at 15ft a) iron banding and b) chert. The red line shows the first low-temperature run and the heating leg of the high-temperature run. The blue line shows the cooling leg of the high-temperature run and the post-heating low-temperature run.

Figure 3.21: Thermomagnetic susceptibility plot that shows the dependence of low-field magnetic susceptibility versus temperature for sample at site G1001 at 43ft a) iron banding and b) chert. The red line shows the first low-temperature run and the heating leg of the high-temperature run. The blue line shows the cooling leg of the high-temperature run and the post-heating low-temperature run.

Figure 3.22: Thermomagnetic susceptibility plot that shows the dependence of low-field magnetic susceptibility versus temperature for sample at site G1001 at 52ft a) iron banding and b) chert. The red line shows the first low-temperature run and the heating leg of the high-temperature run. The blue line shows the cooling leg of the high-temperature run and the post-heating low-temperature run.

Figure 3.23: Thermomagnetic susceptibility plot that shows the dependence of low-field magnetic susceptibility versus temperature for sample at site G1001 at 70ft a) iron banding and b) chert. The red line shows the first low-temperature run and the heating leg of the high-temperature run. The blue line shows the cooling leg of the high-temperature run and the post-heating low-temperature run.

3.4 Magnetic Hysteresis

There were a variety of loops acquired through magnetic hysteresis measurements. The samples from site C27 yielded mainly 'goose-necked' or 'wasped-waisted' loops, indicating multi-domain behavior with high magnetic saturation but low magnetic remanence (figure 3.24). The backfield curves indicate the coercivity of remanence and the sample in figure 3.24(a) has a stronger coercivity of remanence than the sample in figure 3.24(b) indicating the small presence of a hematite as the inflection point is greater than 0.2T and the curve does not reach saturation. Two of the samples, shown in figure 3.25, showed wasp-waisted loops with a larger magnitude of magnetic remanence which is consistent with samples containing both magnetite and hematite (Tauxe et al., 1996).

Figure 3.24: Magnetic hysteresis plots showing a wasp-waisted loop with small magnetic remanence indicating the presence of a low coercivity ferromagnetic mineral (magnetite) in site C27 iron samples at a) 90ft and b) 242ft. The orange lines are backfield remanence curves showing the coercivity of remanence.

Figure 3.25: Magnetic hysteresis plots showing a wasp-waisted loop indicating the presence of both hematite and magnetite in site C27 iron samples at a) 108ft and b) 135ft.

The samples at site C64 contained larger amounts of hematite relative the samples in figure 3.25 as the loops are mostly wasp-waisted with larger ratios of magnetic

remanence to magnetic saturation as shown in figure 3.26. The two samples at 135ft have a stronger narrowing at the waist which means they have more of a low coercivity mineral most likely magnetite. The magnetic saturation is lower compared to the other two sites but the wider waist shows that more of magnetic information could be recorded if not overwritten by another stronger field.

Figure 3.26: Magnetic hysteresis plots showing the larger wasp-waisted loops characteristic of site C64 at a) 130ft iron formation, b) 130ft silica sand, c) 135ft iron formation, and d) 135ft silica sand.

The samples for G1001 are all very similar and are multi-domain grains with little to

no visible remanence figure 3.27. The magnetic saturation is very high for the iron formations and drops drastically for the chert in between indicating a good sorting of ferromagnetic minerals between the layers.

Figure 3.27: Magnetic hysteresis showing the multi-domain loops which characterize all of the samples for site G1001. a) 14ft iron formation, b) 14ft chert, c) 70ft iron formation, d) 70ft chert.

All of the samples were plotted on a Day plot (figure 3.28) with the ratio of magnetic remanence over magnetic saturation (M_r/M_s) versus the coercivity of remanence over coercive force (H_{cr}/H_c). Most of the samples land in the multidomain quadrant

with G1001 and C27 samples making up the all of the multi domain. Only a few samples land within the pseudo-single-domain region with C64 130ft Ri, C27 135 Di and 108 Di, and G1001 43 Di making up the only pseudo-single-domain samples. This is consistent as these samples had some of the strongest Hopkinson peaks in the thermomagnetic curves which would indicate pseudo-single to single-domain grains whereas the other samples have more complex structures mostly with multidomain grains.

Figure 3.28: Day plot showing the ratio of M_r/M_s versus H_{cr}/H_c

3.5 First-Order Reversal Curves (FORCs)

A number of first-order reversal curves (FORCs) were obtained for each site. The first site C27 showed similar FORCs for samples at 70ft and at 230ft to 242ft which were characterized as wasp-waisted to tight multi-domain hysteresis loops. The FORC distribution for the sample at 90ft (figure 3.29) shows a slightly offset center from the x (H_c) axis at 0.075 T as well as being more elongate on the H_c axis extending to 4×10^{-2} T which is consistent with the hysteresis curves showing a stronger magnetic remanence. The multi-domain sample at 242ft (figure 3.30) shows a more central and compact distribution around the origin with the center shifted slightly up at 1×10^{-3} T on the H_u (y) axis and does not extend beyond 10×10^{-3} T on the H_c (x) axis.

The samples that showed a wider wasp-waisted hysteresis curves are exemplified by figure 3.31 which shows a distribution that is shifted off of the H_c axis to about 35×10^{-3} T and shifted down on the H_u axis to -5×10^{-3} T while also being rotated into a 20° tilt with respect to the H_c axis. This distribution is characteristic of two competing ferromagnetic minerals which is consistent with the wasp-waisted character of the hysteresis loop as well as pseudo-single domain indicated by the hysteresis Day plot (figure 3.28). The shift along the H_c axis and the lack of a Verwey transition on the first low-temperature curve of the thermomagnetic analysis (figure 3.13(a))

are consistent with hematite being the main carrier of the magnetic signal with some magnetite causing the elongation along the H_c axis.

Figure 3.29: a) FORC distribution for sample C27 at 90ft iron formation with a smoothing factor of 5. b) The corresponding magnetic hysteresis curve.

Figure 3.30: a) FORC distribution for sample C27 at 242ft iron formation with a smoothing factor of 4. b) The corresponding magnetic hysteresis curve.

Figure 3.31: a) FORC distribution for sample C27 at 108ft iron formation with a smoothing factor of 5. b) The corresponding magnetic hysteresis curve.

The FORC distributions for samples at site C64 were similar for each depth with the FORC distribution shown for depth 135ft in figure 3.32. The center of the distribution is shifted to 5×10^{-3} T along the Hc axis and may be slightly shifted up to 1×10^{-3} T on the Hu axis. The Hc axis reaches to 30×10^{-3} T while the Hu axis reaches a to 25×10^{-3} T and -20×10^{-3} T making the distribution more circular than oblong. The symmetrical aspect and the offset center along the Hc axis indicate little competing forces which would make the sample contain a larger amount of hematite and small amount of magnetite. The samples for the silica sand matrix (figure 3.33) did not group together well but created a general spread along the Hc axis to 0.06 T with a slight tilt at about 20° showing similar structure to figure 3.31 but with a weaker strength and centered closer to the origin.

Figure 3.32: a) FORC distribution for sample C64 at 135ft iron formation with a smoothing factor of 5. b) The corresponding magnetic hysteresis curve.

Figure 3.33: a) FORC distribution for sample C64 at 135ft silica sand with a smoothing factor of 5. b) The corresponding magnetic hysteresis curve.

The samples for G1001 are very similar to that of the deeper samples of C27 (figure 3.30) exhibiting a stretched distribution in the H_u axis and a very small extension along the H_c axis (figure 3.34). The magnetic response is strong and centered very close to the origin shifted slightly up to 1×10^{-3} T on the H_u axis. The chert formation has a weaker response but a similar shape with a little less stretching along the H_u axis (figure 3.35). These plots are consistent with multidomain grains and the chert formations show a more symmetrical distribution indicating only magnetite where the iron formations show some interaction indicating magnetite and most likely some hematite.

Figure 3.34: a) FORC distribution for sample G1001 at 70ft iron formation with a smoothing factor of 5. b) The corresponding magnetic hysteresis curve.

Figure 3.35: a) FORC distribution for sample G1001 at 70ft chert formation with a smoothing factor of 5. b) The corresponding magnetic hysteresis curve.

3.6 Anisotropy of Magnetic Susceptibility (AMS)

One sample for each depth at each site was analyzed for anisotropy of magnetic susceptibility (AMS) and bulk susceptibility for a total 15 samples. The average of the volume normalized bulk susceptibility for samples at sites G1001 is 1.65 ± 0.568 SI with a median of 1.92 SI, at C64 it is $4.65 \pm 1.66 \times 10^{-3}$ with a median of 4.65×10^{-3} , and at C27 it is $2.56 \pm 2.69 \times 10^{-1}$ SI with a median of 1.12×10^{-1} SI. Site G1001 has very high bulk susceptibility while the other two sites also exhibit high susceptibility that is indicative of ferromagnetic minerals such as magnetite and hematite.

Name	Km (SI)	L	F	Pj	T	K1Dec(°)	K1Inc(°)	K2Dec(°)	K2Inc(°)	K3Dec(°)	K3Inc(°)
G1001_14	2.35E+00	1.024	1.41	1.508	0.873	220.4	75.7	69.1	12.6	337.7	6.6
G1001_15	1.94E+00	1.083	1.329	1.467	0.561	54.6	68.5	254.3	20.4	161.8	6.7
G1001_43	1.92E+00	1.066	1.277	1.384	0.588	338	22.4	185.9	65	72.4	10.5
G1001_52	1.32E+00	1.089	1.702	1.952	0.723	69.3	62.1	268.2	26.6	174.3	7.8
G1001_70	7.26E-01	1.226	3.532	4.893	0.722	347.3	64.8	151.7	24.4	244.4	6
Averages	1.65E+00	1.10E+00	1.85E+00	2.24E+00	6.93E-01	2.06E+02	5.87E+01	1.86E+02	2.98E+01	1.98E+02	7.52E+00
Median	1.915	1.08E+00	1.41E+00	1.51E+00	7.22E-01	2.20E+02	6.48E+01	1.86E+02	2.44E+01	1.74E+02	6.70E+00
St Dev	5.68E-01	6.81E-02	8.54E-01	1.34E+00	1.12E-01	1.26E+02	1.87E+01	7.25E+01	1.82E+01	8.87E+01	1.60E+00
C64_130	3.00E-03	1.004	1.001	1.006	-0.575	114	70.5	6.4	6.1	274.4	18.4
C64_135	6.31E-03	1.051	1.033	1.086	-0.208	4.6	45.6	148	38.2	253.8	19.2
Averages	4.65E-03	1.03E+00	1.02E+00	1.05E+00	-3.92E-01	5.93E+01	5.81E+01	7.72E+01	2.22E+01	2.64E+02	1.88E+01
Median	4.65E-03	1.03E+00	1.02E+00	1.05E+00	-3.92E-01	5.93E+01	5.81E+01	7.72E+01	2.22E+01	2.64E+02	1.88E+01
St Dev	1.66E-03	2.35E-02	1.60E-02	4.00E-02	1.84E-01	5.47E+01	1.25E+01	7.08E+01	1.61E+01	1.03E+01	4.00E-01
C27_70	5.81E-01	1.225	1.11	1.366	-0.321	31	18.3	297.9	9.2	182.6	69.4
C27_90	2.71E-02	1.128	1.174	1.326	0.142	144	26.9	44.4	18.1	284.6	56.8
C27_108	1.09E-02	1.034	1.148	1.2	0.611	157.3	23.3	40.3	46.5	264.4	34.2
C27_125	1.23E-01	1.097	1.258	1.394	0.424	327.6	3.8	233.4	47.8	61.1	42
C27_135	1.14E-02	1.063	1.195	1.282	0.49	254.5	11.7	162.6	8.9	36.2	75.2
C27_230	1.02E-01	1.192	1.156	1.379	-0.095	173	19.4	67.3	37.5	284.4	46.2
C27_240	4.84E-01	1.192	1.243	1.483	0.106	178.6	11	275.9	33.3	72.8	54.5
C27_242	7.09E-01	1.237	1.301	1.61	0.106	157.6	3.8	248.6	14.2	52.8	75.2
Averages	0.25607	1.15E+00	1.20E+00	1.38E+00	1.83E-01	1.78E+02	1.48E+01	1.71E+02	2.69E+01	1.55E+02	5.67E+01
Median	0.11245	1.16E+00	1.18E+00	1.37E+00	1.24E-01	1.65E+02	1.50E+01	1.98E+02	2.57E+01	1.28E+02	5.57E+01
St Dev	2.69E-01	7.15E-02	6.00E-02	1.17E-01	2.92E-01	8.05E+01	8.05E+00	1.01E+02	1.52E+01	1.04E+02	1.45E+01

Table 3.2

Summary of the anisotropy of magnetic susceptibility data for sites C27, C64, and G1001 with averages, medians, and standard deviations. Km: Mean magnetic susceptibility (SI); L: Magnetic lineation; F: Magnetic foliation; Pj: Corrected degree of anisotropy; T: Shape parameter; K1, K2, K3: Maximum, intermediate, and minimum magnetic susceptibility axes in declination (Dec) and inclination (Inc) degrees.

A Jelinek plot was created for each of the sites which is a plot of the T (shape parameter) versus Pj (corrected degree of anisotropy) which characterizes the anisotropy as either an oblate or prolate shape (Jelinek, 1981). The samples from site C27 mostly exhibit a oblate shape that is consistent with most BIF samples (Table 3.2). The samples at 108ft, 125ft, and 135ft show consistent values of T between 0.5 and 1, whereas the samples at 90ft, 240ft, and 242ft show lower levels of oblateness. However, the samples at 70ft and 230ft have a prolate shape anisotropy, inconsistent with strong banded ferromagnetic minerals typical in banded iron formations (figure 3.36). The positive correlation between the mean bulk susceptibility and the degree of anisotropy is weak with $R^2 = 0.6497$.

Figure 3.36: a) Jelinek plot showing the shape of the anisotropy to be oblate. b) Mean Bulk Susceptibility versus corrected degree of anisotropy showing the R^2 correlation factor.

The samples from site 64 are both characterized by prolate anisotropy. However, they also exhibit inflection points at low temperatures on their cooling $\kappa(T)$ curves, which indicates ferromagnetic clays whose presence could explain the behavior atypical for

BIFs. Since there are only two samples from this site, no correlation between the corrected degree of anisotropy and the mean bulk susceptibility can be inferred.

Figure 3.37: a) Jelinek plot showing the shape of the anisotropy to be oblate. b) Mean bulk magnetic susceptibility versus corrected degree of anisotropy. No correlation factor is calculated since there are only two points.

All samples from site G1001 have oblate anisotropy, with the values of T varying between 0.5 and 1 (Table 3.2), thus exhibiting typical BIF characteristics. The correlation factor between the corrected degree of anisotropy and mean bulk susceptibility is the highest at 0.7626 but shows a decreasing correlation of the degree of anisotropy as the mean bulk susceptibility increases. This could be due to the large degree of folding and as more ferromagnetic minerals are introduced there will be a higher chance that the minerals will have been shifted or folded to point in a different direction.

Figure 3.38: a) Jelinek plot showing the shape of the anisotropy to be oblate. b) Mean Bulk Susceptibility versus corrected degree of anisotropy showing the R^2 correlation factor.

All the samples were plotted on equal area plots as shown in figure 3.39. There are few locations where the minimum susceptibility axis (blue circle) is directly next to a maximum susceptibility axis (red square) which suggests an inverse fabric indicating single domain grains. This is consistent with the Day plots from the magnetic hysteresis analysis.

Figure 3.39: Equal area projections on the lower hemisphere for a) site C27 b) site C64 and c) site G1001 where K1 (max) are red squares, K2 (intermediate) green triangles and K3 (min) are blue circles.

3.7 Königsberger Ratio

The Königsberger ratios (Q) were calculated and compared between the sites which show a pretty wide range of Q values (3.40). Most of the shallow samples for site C27 display a $Q < 1$ with a stronger induced magnetization. Most of the deeper sites for C27, C64 at 130ft, and most of the samples for G1001 display a $Q > 1$ indicating a stronger NRM component.

Figure 3.40: Induced magnetization (J_i) versus remanent magnetization (J_{nrm}) with the dashed line showing the Königsberger ratios (Q) = 1 for the three sites (C27, C64, and G1001).

Names	J _{nrm} (A/m)	k(Si)	H (A/m)	J _i (A/m)	Q
C27_70	4.69E+00	5.81E-01	43.8	2.54E+01	1.84E-01
C27_90	6.89E-01	2.71E-02	43.8	1.19E+00	5.80E-01
C27_108	1.62E-01	1.09E-02	43.8	4.75E-01	3.41E-01
C27_125	8.80E-01	1.23E-01	43.8	5.37E+00	1.64E-01
C27_135	2.01E-01	1.14E-02	43.8	4.98E-01	4.04E-01
C27_230	1.95E+00	1.02E-01	43.8	4.48E+00	4.36E-01
C27_240	1.34E+02	4.84E-01	43.8	2.12E+01	6.32E+00
C27_242	5.03E+01	7.09E-01	43.8	3.10E+01	1.62E+00
C64_130	5.49E+00	3.00E-03	43.8	1.31E-01	4.19E+01
C64_135	2.93E-01	6.31E-03	43.8	2.76E-01	1.06E+00
G1001_14	1.79E+02	2.35E+00	43.8	1.03E+02	1.74E+00
G1001_15	1.60E+02	1.94E+00	43.8	8.50E+01	1.88E+00
G1001_43	1.62E+02	1.92E+00	43.8	8.38E+01	1.93E+00
G1001_52	1.11E+02	1.32E+00	43.8	5.78E+01	1.92E+00
G1001_70	3.63E+00	7.26E-01	43.8	3.18E+01	1.14E-01

Table 3.3

Samples and their NRM component (J_{nrm} , the bulk susceptibility (κ), the induced magnetization component (J_i , and the resulting Königsberger ratio (Q).

Figure 3.41: Bar plot showing Q values for each sample excluding C64 at 130ft. C64 at 130ft has a Q value of 40 and skews the plot.

3.8 Scanning Electron Microscopy

Scanning electron microscopy can be used to directly observe the shape and orientation of the mineral grains with backscatter electron (BSE) images and secondary electron (SE) images as well as to determine the presence of certain elements through energy dispersive spectroscopy (EDS). All these methods were used to investigate a sample from each of the sites. Figure 3.42 presents the polished samples before being coated in carbon. Samples for G1001 and C27 at 125 ft depth show distinct banding between dark and light greys while the sample at 135 ft exhibits more granular features with some reddish coloring. The sample for C64 shows the stark contrast between large iron inclusions and the red silica sand matrix in between.

Figure 3.42: SEM samples after polishing and before coating.

Two samples were analyzed for site C27. The first is at 125 ft and shows the iron bands and the surrounding matrix. An overview of the banding is shown in the BSE image in figure 3.43. The image shows the iron banding is not quite uniform and contains some of the silicates mixed in with the iron. A closer image of the iron formation is shown in figure 3.44 with spectra acquired at three spots within the sample. Spectra 137 and 138 are not shown as they are almost exactly the same as spectrum 136. Spectrum 135 shows the large amount of iron and oxygen that

makes up the iron bandings where spectrum 136 shows the high level of silica that makes up the silicate matrix. Spectrum 134 shows multiple light elements such as calcium, sodium, manganese, aluminum, silicate, potassium with a small amount of iron as well. An EDS map was obtained over a similar area (figure 3.45), which shows a backscatter image and the relative abundances of different elements found within that image. The iron matches up nicely with the bright spots and silica is most abundant in the darkest of regions. Aluminum matches up with sodium and potassium in different areas. Spectrum 134 from figure 3.44(a) could be on one of the interlapping areas. The potassium aluminum silicate could be an orthoclase while the sodium aluminum silicate could be a form of oligoclase. The magnesium calcium and iron locations are possibly actinolite, $Ca_2(Mg_{4.5-2.5}Fe_{0.5-2.5}^{2+})Si_8O_{22}(OH)_2$.

Figure 3.43: Backscatter electron image of C27 at 125ft showing a zoomed out portion. Brighter spots are heavier elements and correspond to the presence of iron where the darker spots are silicates.

Figure 3.44: a) BSE image showing a closer examination of the iron banding. b) Spectra taken from the spots in the BSE image in a).

Figure 3.45: EDS maps showing the relative abundance (the brighter the color the greater abundance) of each element in the BSE image. a) BSE image b) iron, c) silica, d) aluminum, e) sodium, f) potassium, g) magnesium, and h) calcium.

The next sample from C27 was from 135ft and the overview BSE image shows very little banding and a well mixed matrix of iron and silicates (figure 3.46). A closer view of the matrix is shown in figure 3.47 with the accompanying spectra that characterize

the major regions obtained. Spectrum 169 shows the EDS of the bright pixels to contain a mixture of iron with a little manganese. Spectrum 165 gives the spectra for the silicates bearing a small enough amount of iron that it might be a contamination from the iron surrounding the silicate. Spectrum 170 is interesting because while taken from the same grain as spectrum 169, it shows considerably less iron and a wider range of elements such as potassium, aluminum, magnesium, and a large amount of silica. This is most likely an iron rich clay supported by the thermomagnetic curve (figure 3.15).

Figure 3.46: BSE image showing the well mixed composition of iron and silicates.

Figure 3.47: a) BSE image showing a closer view of the matrix in the sample for C27 at 135 ft. b) Unique EDS spectra obtained for this image.

A SE image was taken looking into one of the holes in the sample and while most of the images looked similar to the backscatter image in figure 3.47 with flat sheetlike structures there were some blocky cubic structures similar to the ones shown in figure 3.48.

Figure 3.48: SE image inside of a hole in the polished sample showing the blocky structure of some of the silicates.

The sample from C64 showed a large homogeneous block of iron within a silica sand matrix similar to what was seen with the naked eye (figure 3.49). Figure 3.50 shows a zoomed-in image of the iron body and the corresponding spectra. Both spectra indicate a small amount of manganese within the iron. The very small peaks of Al and Si that disappear from spectrum 90 to 91 could be indicative of leftover manganese enriched grunerite, $(Mn_2^{+2})(Fe_5^{2+})(Si_8O_{22})(OH)_2$. The sand silica matrix is analyzed in figure 3.51 which shows the simple silica sand in spectrum 96 and a potassium aluminum silicate filled in between the sand grains in spectrum 97 that corresponds to orthoclase, $KAlSi_3O_8$. The dark areas mapped by spectrum 98 and 100 are the epoxy that the sample was placed in to be held together for polishing. Spectrum 99

shows there are some iron manganese oxides that are in the silica sand matrix that also seem to be mostly in the potassium aluminum silicate filling.

Figure 3.49: A BSE image of the C64 sample showing a zoomed-out image of the homogeneous iron body (light grey) with the silica sand matrix on the top of the image (dark grey).

Figure 3.50: a) A BSE image of the C64 sample showing the iron body, and b) the EDS spectra acquired from the homogeneous iron body.

Figure 3.51: a) A BSE image of the 64 sample showing the silicate sand matrix. b) The corresponding EDS spectra.

Unlike the samples for site C27, there were a few instances of heavier metals and some rare earth elements (REE) observed. Figure 3.52 shows one of the instances of an iron-nickel sulfide. The amount of nickel is small but sufficient to be registered as brighter pixels within the iron body. More instances of these small iron-nickel sulfides were found than any of the other heavier metals but still they constituted a very small amount within the iron body. For example, another mineral inclusion within iron was found to contain zirconium (figure 3.53) but very few sites were found to contain zirconium in comparison to the iron-nickel sulfide. In addition, a very small number of inclusions (bright spots) that contained REEs was identified (figure 3.54). Spectrums 115 and 116 show elements gadolinium and ytterbium with trace amounts of dysprosium.

Figure 3.52: a) A BSE image of the C64 showing two brighter inclusions within the iron matrix. b) Spectra showing that both inclusions are an iron-nickel sulfide.

Figure 3.53: a) A BSE image of the C64 sample showing another bright inclusion within the iron body. b) The corresponding spectrum showing a large amount of zirconium.

Figure 3.54: a) A BSE image of the C64 sample showing a bright spot within iron body. b) Spectra showing that the bright spot contains rare earth elements.

The sample for G1001 is shown in a low-magnification BSE image that demonstrates

the well-defined banding, which is consistent with initial appraisal by visual inspection (figure 3.55). Electron dispersive spectral mapping for iron, silica, magnesium and manganese was conducted over the area shown in the overview BSE image (figure 3.56). The iron bands (figure 3.56(a)) are distinct from the silica bands (figure 3.56(b)), with both magnesium and manganese primarily concentrated in the silicates (figure 3.56(c) and 3.56(d)). EDS analyses further indicate the presence of iron-rich and silica-rich compositions spectrum as well as those enriched in Mg, Mn, and REE (figure 3.57). Spectrum 158 shows a homogeneous iron oxide with no trace of any other element, which is consistent with the FORC diagrams showing no competing ferromagnetic minerals. The silicate matrix has two different types. The first type is a completely silica-rich matrix exemplified by spectrum 153. The iron rich mineral is most likely magnetite and the silica-rich mineral is most likely chert. The second type of silica rich mineral contains aluminum, calcium, magnesium, and manganese along with small amounts of iron as shown in spectrum 152 which is most likely a hornblende amphibole. The REE shown in spectrum 151 are cerium, lanthanum, and neodymium. Similar amounts of these REEs are also found in other bright spots throughout the iron bands.

Figure 3.55: A low-magnification BSE image of the G1001 sample showing distinct and well sorted banding. Brighter and darker colors show iron-rich and silica-rich bands, respectively.

Figure 3.56: Elemental EDS maps that correspond to the BSE image in figure 3.55 with a brighter color indicating a greater abundance of the element: a) iron, b) silica, c) magnesium, and d) manganese.

Figure 3.57: a) BSE image showing a wide array of compositions including iron (intermediate grey; spectrum 158), the silicate matrix (dark grey; spectrum 153), and minerals enriched in magnesium, manganese, and REE (light grey; spectra 151 and 152). b) Characteristic spectra for each of these compositions.

Discussion

The results of rock magnetic characterization of the three sites investigated in this project indicate the presence of two clearly distinct iron formations exemplified most notably by the well-laminated samples collected from site G1001 and the granular formation at site C64. The deeper samples from site C27, 230ft to 242 ft, are most similar to the well-laminated samples from site G1001, whereas the shallower samples from site 27 demonstrate an increase in the hematite content but not to a composition that is similar to the samples from site C64. All three of the site's samples exhibit a clear difference in appearance (texture) and each particular appearance tends to correspond to similarities in magnetic characteristics. The samples that are characterized by granular texture tend to contain a larger amount of hematite (for example, the samples from site C64 and several samples from site C27 such as those at 108 ft and 135 ft (figures 3.25(b) and 3.46). The more tightly banded samples as exemplified by those in G1001 have almost entirely stoichiometric magnetite as their magnetic mineral (figures 3.55 and 3.34(b)). This correlates strongly with the geologic maps shown in figures 1.3 and 1.4 that display the investigated cores cutting through the fault zone (possibly with Felch formation) and Vulcan iron formations respectively.

There have been instances of the Vulcan Iron Formation being separated into a younger (Curry member) and older (Traders member) (James et al. 1961 and Cumberlidge and Stone 1964). The upper Vulcan formation is typically more granular or oolitic and hematitic than the Traders member, which is typified by well-banded magnetite (Cumberlidge and Stone, 1964).

The deepest samples from site C27, 230ft to 242ft as well as the shallowest at 70 ft, are well characterized by the description of the lower Vulcan iron formation, as are all of the samples collected from site G1001. The narrow structure of the hysteresis loops, quick saturation of the backfield dc curves, and the well centered, vertically oblong FORCs are all emblematic of stoichiometric magnetite (figure 3.30 for site C27 and 3.34 for site G1001). Much of the initial NRM component was lost during the low temperature demagnetization (figure 3.4), which necessitates a strong Verwey transition that is demonstrative of these samples (figures 3.17 and 3.19), as well as over 95% of the NRM component lost after heating to 600°C in an oven (figure 3.5). Most of these lower Vulcan formation samples display Königsberger with a $Q > 1$ indicating the NRM component is dominant over the induced magnetization components.

According to the interpretation by James et al. (1961), the Michigamme Slate is located between the shallow and deep samples at site C27 which indicates the Vulcan formation has been overturned. Initially looking at figure 1.4(b) would intimate the deeper

core corresponds to the upper Vulcan formation. This was not the case as the deeper samples correspond to the lower Vulcan Formation. Although it would explain why the sample at a depth of 70 ft is more characteristic of the lower formation. Another explanation is the sample at 70 ft is right at the border of the fault zone shown in figure 1.4(b) and could have been displaced to this location. The metamorphic process of folding does explain the large quartz grains found at 125 ft and the poor banding in the samples from 90 ft to 135 ft.

The shallower sections of C27 tend to show a presence of hematite apart from the shallowest sample at 70ft that closely resembles the magnetite-bearing lower Vulcan formation. The samples from 90 ft to 35 ft exhibit varying amounts of hematite through slightly wider waists in the hysteresis loops, inflection points at high field values on the backfield curves, and stretched distributions on the FORC diagrams suggesting a much stronger hematite to magnetite ratio (figures 3.31 and 3.31). The samples lose very little of their NRM component during the low temperature demagnetization (figure 3.4) that is correlated with their almost non-existent Verwey transition during the first low temperature thermomagnetic curve (figure 3.13). In addition, except for the sample at 90 ft, the samples lose very little of their NRM component after heating to 600°C (figure 3.5). The magnetite to hematite ratio is a strong indicator of the upper or lower member of the Vulcan iron formation which is consistent with previous magnetic characterization of the formations based on the surface outcrop samples (Laird, 2017). The Königsberger ratios for the shallower

section display a $Q < 1$ indicating the induced magnetization component is more dominant than the NRM component.

The samples from C64 are characteristically strong in hematite showing much wider waists in hysteresis loops, stronger fields necessary to reach saturation for backfield curves, and FORC diagram distributions offset from the horizontal axis (figure 3.32). The thermomagnetic curves indicate a much weaker initial Verwey transition (figure 3.18) that is reflected in the lower percentage of NRM removed by the low temperature demagnetization (figure 3.4). However, only the sample from 135 ft retained a decent portion of the NRM after heating to 600°C (figure 3.5). These characteristics are all in line with the heavily oxidized quartz-mica sections of the Felch formation which could be what was captured in the fault zone. The Königsberger ratios display a $Q \approx 1$ at 135 ft and $Q \gg 1$ at 130 ft. This disparity between the Q values is interesting as the other samples that contained larger amounts of hematite tended to have ratios of $Q < 1$.

According to the data the samples from site G1001 as well as the deepest samples from site C27, including the sample from 70 ft, are consistent with the older lower Vulcan formation. The samples taken from a shallower depth from site C27, 90 ft to 135 ft, are more consistent with the upper Vulcan formation member as hematite starts to dominate the magnetic characteristics. The surface samples taken for previous magnetic characterization were taken south of the location where the drill sites are

located (figure 1.5). The shallow samples characterized as the upper Vulcan formation from site C27 are similar to those characterized in the previous work as the Curry member (Laird, 2017). Some of the Curry members characterized in the previous work have some similarities to the granular samples from site C64 as do the samples from 90 ft to 135 ft from site C27. Wide waisted hysteresis loops and FORCs offset from the horizontal axis with a slight downward tilt as well as thermomagnetic curves showing the transition of iron bearing clays to magnetite with some small conversion of hematite. The Curry members exhibit little NRM component removal as a percentage after both LTD and temperature treatment similar to the shallow samples from site C27 (Laird, 2017). The samples have similar magnetic characterization and appear to have similar mineral composition.

The comparison to the Traders member does not match quite as well. The surface samples seem to contain more hematite than the core samples from G1001 although they do match more closely to some of the samples from C27 from depths 90 ft to 135 ft that present small amounts of hematite. The surface samples taken from the Traders member contain larger amounts of hematite when compared to the samples from site G1001 and the deepest samples from C27. The core samples indicate very little to no hematite and seem to be dominated entirely by magnetite whereas the surface Trader samples exhibit slightly wider waisted hysteresis loops, and less magnetic stability as well as the presence of hematite in the thermomagnetic curves. The differences may be due to discrepancy in grain sizes as the core samples contain multidomain to

pseudo-single domain grains in the Day plot (figure 3.28) and the thermomagnetic curves (figures 3.19) whereas the surface samples contained more pseudo-single to single domain grains (Laird, 2017). The difference may also be explained due to weathering of the surface samples and the oxidation of magnetite to hematite. This is also consistent with the discrepancy between bulk susceptibilities of the core to surface samples with the core samples one to two orders of magnitude stronger than the surface samples (Laird, 2017).

There were a wide range of Königsberger ratios indicating the magnetite heavy lower Vulcan formation was dominated by the NRM component while the upper Vulcan formation containing more hematite was dominated by the induced magnetization component (figure 3.40). The differences in the formations played a larger role in determining the Q ratio rather than changes in depth. This is further advanced as the deepest core samples from C27 are very similar to very shallow samples at G1001 where the type of formation plays a larger role in determining the magnetic characterization than the depth of the formation.

The wide range of Q values demonstrating the range between induced magnetization and NRM appears to be tied to the different members and formations which can be applied to the interpretation of USGS aeromagnetic survey data. The anisotropy of magnetic susceptibility parameters indicating the major and minor axis (K1 and K3

respectively) will also be useful for correction and analysis of the obtained survey data.

Conclusion

Core samples from three sites, two of the Vulcan iron formation and one from a fault zone indicative the Felch formation, were magnetically characterized. Magnetic characterization was able to separate the Vulcan formations into two distinct types based on the ratio of magnetite to hematite with the lower (older) members dominated by magnetite and the shallow (younger) members shown to have more hematite that interacts with magnetite. The faulted zone samples were shown to be dominated almost entirely by hematite with very little magnetite. The shallow Vulcan iron formation is similar to the surface samples magnetically characterized in a region a few miles south. The deeper formation shows strong similarities but the core samples are shown to be almost entirely magnetite while the surface samples contained some amounts of hematite in addition to magnetite. The lack of hematite in the core samples may explain the increase in magnetic susceptibility values between the core samples and surface samples which could also account for the strong magnetic signals in the aeromagnetic survey that were not explained with the surface samples. The magnetic characterizations were linked more closely to the different members and formations than to the depth that the samples were acquired. The anisotropy of

magnetic susceptibility and Königsberger ratio data will be useful for corrections needed for a USGS aeromagnetic survey conducted over the area.

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